

**THERMOPHYSICAL ESEM & TEM
CHARACTERIZATION of CARBON FIBERS
CTE, SPECTROSCOPY and ROUGHNESS STUDIES at
HIGH TEMPERATURES
(Grant NAG 8 –1802)**

TEES Project Number: 64350

Project Report

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Executive Summary

Accurate determination of the transverse properties of carbon fibers is important for assessment and prediction of local material as well as global structural response of composite components. However the measurements are extremely difficult due to the very small diameters of the fibers (few microns only) and must be conducted within a microscope. In this work, environmental scanning electron microscope (ESEM) and transmission electron microscope (TEM) are used to determine the transverse coefficient of thermal expansion of different carbon fibers as a function of temperature.

In ESEM, images of the cross section of the fiber are taken at room temperature and at various elevated temperatures, the change in the perimeter (diameter) of the fiber is used to evaluate the transverse coefficient of thermal expansion (TCTE), as presented in equation (1)

$$\alpha_T = \frac{D_2 - D_1}{D_1(T_2 - T_1)} = \frac{\Delta D}{D_1 \Delta T} \quad (1)$$

The fibers are subjected to 3 - 4 heating cycles from room temperature to 850°C and they are held at each temperature step for about 6 minutes before taking images to allow them to stabilize.

Different tests are performed to gain insight to the features inherent in the fibers as below summarized briefly

1. The crystallinity (ordering of the graphic basal planes) of the fibers was analyzed by X - ray spectroscopy in TEM. Crystallinity was examined for fibers in the as received condition and also after subjecting them to a heat treatment cycle. For example, the skin and core area could be easily distinguished in the NASA - T1000 fibers. In the as received fibers the skin possessed more graphic ordering than the core area. After subjecting these fibers to three cycles of heating to 800°C, the ordering of the graphic planes in the skin markedly increased whereas minimal change occurred in the core. The NASA -

T300 fibers were heat treated at 2500°C before we received them. Their graphic planes in the skin as well as the core were aligned and remained so after exposure in the tests. CTE was measured for the as received and heat treated fibers and as expected the heat treated samples with more graphic ordering had lower CTE values.

2. Energy-Dispersive X-ray Spectrometry (EDS) and Wavelength-Dispersive X-ray Spectrometry (WDS) were conducted to find out the elemental composition of the carbon fibers. These tests revealed that the fibers primarily contained carbon but also had some traces of nitrogen and in the case of AMOCO-P55J2K sulfur was noted.
3. The surface roughness of the fibers was documented in SEM. Three different types of surface finishes were observed, namely smooth, lines running parallel to the fiber axis and sizing on the fiber.

Different testing methods were also developed to get better results from ESEM studies:

1. Fiber slicing method: The fibers were embedded in epoxy and thin slices around 2μ in size were cut on the microtome. These slices were mounted on a circular plate in the ESEM and the epoxy was burned off by using the heating capabilities of the ESEM. This method ensured better positional stability.
2. A hole of 0.7mm was drilled in a graphite disc of diameter 5mm and thickness 3mm. About fifty fibers were press fit into the hole by using a pencil lead. This prevented the fibers from moving laterally.

For the temperature range under consideration, the change in the diameter of the fiber is around 10nm – 20nm. The resolution of the ESEM is 5nm and hence the change in the diameter of the fiber image on the computer is about 2 – 4 pixels. This is very hard to detect and hence the testing is shifted from ESEM to TEM equipped with heating stage, which has a guaranteed resolution of 0.23nm.

The fibers of length 2.5mm are mounted on an ellipsoidal washer and a similar washer is placed on top of the fibers for the TEM observations. This assembly is secured in the heating stage by a clamping screw. The images of the fiber are taken at room temperature and at 850°C on negatives, which are then digitized at high resolution (4000 dpi) and analyzed using Adobe Photoshop. The diameter of the fibers is read off at corresponding locations in the image at room temperature and 850°C and the change in diameter is used for evaluating CTE as per equation (1). Multiple readings for the diameter are taken from the same set of images and the average CTE and the standard deviation is evaluated for a fiber. With the higher resolution of the TEM, the CTE values obtained were considerably lower in magnitude as well as in scatter leading us to conclude that TEM indeed assures us of realistic values.

The report is presented in the chronological order of our findings and improvements to our techniques including the rationale of designing apparatus and interpreting the results from digitized images. As a summary, the TEM based transverse CTE values for the fibers in the study are presented in Table 1.

Table 1: Transverse CTE for fibers studied

Fiber	Average Transverse CTE @ 400°C	Average Transverse CTE @ 800°C	Average Transverse CTE @ 850°C	Average Transverse CTE @ 1100°C
IM7	-	-	4.58E-06	-
IM7	-	-	4.21E-06	-
IM7	-	-	4.86E-06	-
IM7	-	-	5.13E-06	-
T-1000-HTT-01	-	-	5.28E-06	-
T-1000-HTT-01	-	-	1.16E-05	-
T-1000-HTT-01	-	-	2.80E-06	-
T-1000-HTT-01	-	-	8.03E-06	-
NASA T1000HT02	1.58E-06	2.31E-06	-	-
P55	-	-	3.63E-06	-
P55	-	-	3.35E-06	-
P55	-	-	3.28E-06	-
Hi Nicalon Type S2	8.75E-07	1.80E-06	-	-
Hi Nicalon Type S2	2.28E-06	2.82E-06	-	3.70E-06

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❖ Accomplishments

1. Monthly Summary

Week of	
April 2	<ul style="list-style-type: none">• Implemented the image processing techniques that Dr. Ray Guileemette of electron microprobe lab had taught us to improve our measurements.• Updated fiber data to include new fibers sent by AMOCO and BF Goodrich Aerospace• Ordered, received and installed the digitizing tablet for improved measurements• Completed ESEM Test (holding time, 1 cycle, T-300)• Calculated perimeter of T-300 data using the latest technique
April 9	<ul style="list-style-type: none">• Recalculated perimeter of all T300 data to date• Calculated thermal strain and CTE as a function of temperature• Reviewed the thermal expansion of anisotropic fibers using Elasticity
April 16	<ul style="list-style-type: none">• Conducted Library search of papers of Z. Hashin• Started training to use ESEM independently of technicians• Read papers discussing other CTE measurement techniques
April 23	<ul style="list-style-type: none">• Studied papers related to CTE and measurement technique• Started simple models of thermal expansion of anisotropic fiber using Elasticity theory.• Continued to analyze the T-300 strain and CTE data and presented comparisons in tables and graphs

2. Focus Areas

- Searched for the suitable definition of CTE for our data; perimeter vs area changes and their relevant impact
- Tested T-300 fiber in ESEM by holding each temperature for 15min. and evaluated the corresponding perimeter, strain and CTE.
- Jiwoong trained to use ESEM machine for 8 hours so he can use it after hours without a technician.
- Analyzed and compared the data from single cycle holding time to data obtained from three continuous cycles without holding time.
- Developing an understanding for the mechanism of thermal expansion in anisotropic fibers based on elasticity theory.

3. Results

Following two graphs depict the CTE calculations from the results of ESEM tests for T-300 fiber. The result of unholding time test conducted over three cycles is presented in Figure 1. When 15 minutes of holding time was utilized at each 100C increment, the results became those displayed in Figure 2. Note that there is a distinct band in both cases. We are making progress in understanding how to calculate a reasonable CTE from digitized perimeters.

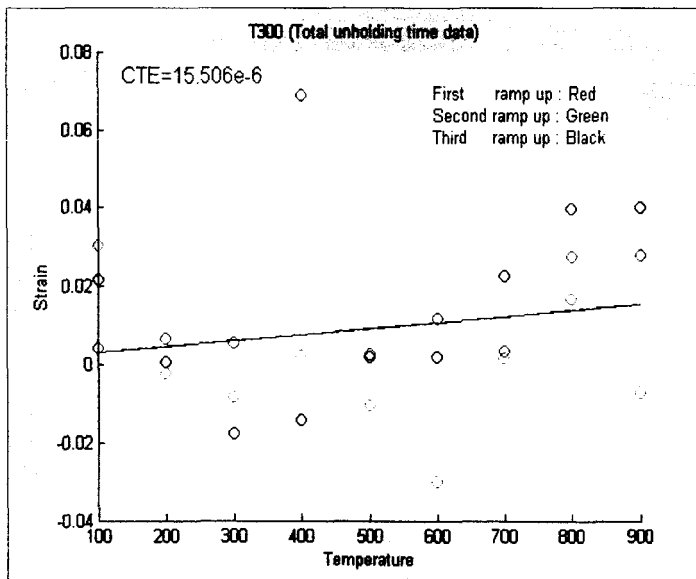


Fig. 1 CTE in three cycles w/o holding time

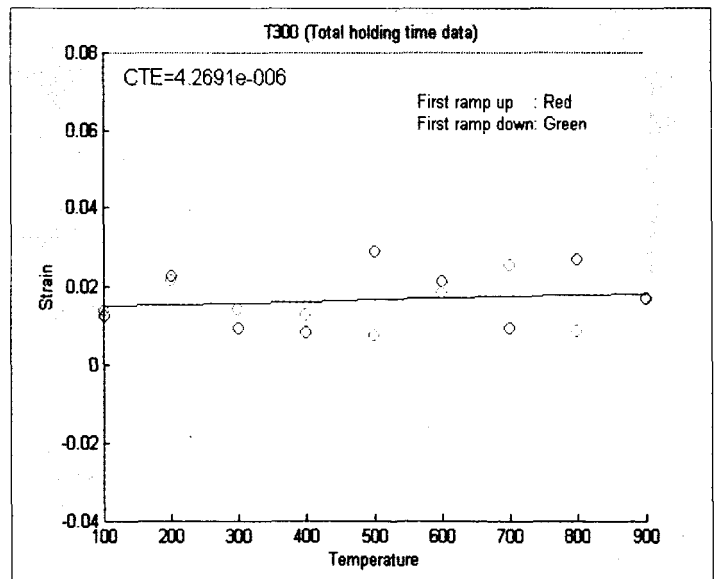


Fig. 2 CTE in one cycle with holding

4. Future work

- Test T-300 by holding time and 3 cycles and will analyze the data.
- Continue to research the mechanism of thermal expansion for anisotropic fiber
- Implement statistical evaluation of the current images.

List of fibers

specimen	Treat	CO.	Piece
AS4			1(short)
T1000			1(long)
T900			1(long)
T650			1(long)
T300			1(long)
P55			1(long)
IM8			1(long)
IM7			1(long)
M30S-TORAY			1(long)
INCO			a little
FOAM		AFRL	3~4
T300	Heat treated after rigidization	BF Goodrich aerospace	6~7
T300	Heat treated after PYC	BF Goodrich aerospace	4~5
T300	Pan based	AMOCO	1 box
T650	Pan based	AMOCO	1 box
P55J2K	Pitch based	AMOCO	1 box

List of papers

1. Wagoner, G., Smith, R.E., and Bacon, F., "Thermal Expansion and Elastic constant Measurements of Carbon Fibers," in Extended Abstracts of 18th Biennial Carbon Conference, Worcester Polytechnic Institute, MA, pp. 415-416, 1987.
2. Wagoner, G. and Bacon, F., "Elastic constant and Thermal Expansion Coefficients of Various Carbon Fibers," in Extended Abstracts of 19th Biennial Carbon Conference, Penn State University, PA, pp. 296-297, 1989.
3. Marciniak, W. and Rozploch, F., "Measurement of the Radial Thermal Expansion Coefficient of Carbon Fibers," High Temperatures-High Pressures, v11(1979) 709-710
4. Rozploch, F. and Marciniak, W." Radial Thermal Expansion of Carbon Fibers," High Temperatures-High Pressures, v18(1986) 585-587
5. Sheaffer, P. M., "Transverse Thermal Expansion of Carbon Fibers," in Extended Abstracts of 18th Biennial Carbon Conference, Worcester Polytechnic Institute, MA, pp. 20-21, 1987.
6. Van Schooneveld, H.G. and Diefendorf, R.J., "Transverse Thermal Expansion in Carbon Fibers and their Composites," in Proceeding of the Baden-Baden Carbon Conference, West Germany, p.606, 1980
7. Fanning, R.C. and Fleck, J.N., "Transverse Thermal Expansion Characteristics of Graphite Fibers" in Extended Abstracts of 10th Biennial Carbon Conference, Lehigh University, PA, pp. 47-48, 1971.
8. Z Hashin, B.W. Rosen and R.B. Pipes, Nonlinear effects on composite laminate thermal expansion

Book

1. K.H.G. Ashbee, "Fundamental Principles of Fiber Reinforced Composites"
2. Mechanics of Composite Materials : Recent Advances / edited by Zvi Hashin, Carl T. Herakovich.

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Accomplishments

1. Monthly Summary

Week of	
May 7 May 14	<ul style="list-style-type: none">• Final exams• Out of town
May 21	<ul style="list-style-type: none">• Statistical evaluation for the following T-300 data: 3 cycles w/o holding• Literature review on other CTE measurement techniques.• Received fibers and mini-tow specimens from Rockwell Science Center.
May 28	<ul style="list-style-type: none">• Literature review related to CTE and measurement techniques• Training to use ESEM independently of technicians• Completed ESEM Test (with holding time, 3 cycles, T-300)• Analyzed the T-300 strain and CTE data and presented comparisons in tables and graphs

2. Focus Areas

- Implemented a statistical method to search for a suitable regression curve. Evaluated correlation coefficients for the data set on T-300 fiber (w/o holding time, 3 cycles, T-300)
- Completed the cyclic thermal testing of another T-300 fiber in ESEM by holding the specimen at each temperature for 10 min. during ramp up and cool down (total of 3 cycles) and evaluated the corresponding perimeter, strain and CTE.
- Jiwoong continued his training on using ESEM after hours.

3. Results

- Statistical method

Our studies revealed that a second order polynomial function is most suitable to data obtained from the experiment w/o holding time, 3 cycles for T-300. We omitted the outlier data at T=400C based on comparisons of P-value. Each P-value for the coefficients of polynomial function must be less than 0.05 to be a suitable regression function. Overall, first ramp up data and third ramp up data well correlated within this data sets.

b) Evaluation of circumferential CTE

The result of holding time test conducted over two cycles is presented in Figure 1 where both ramp up and ramp down data are included. This data is obtained from the latest experiment where three cycles were utilized. However, during the first ramp up/ramp down, the fiber that was being observed was distorted to the view angle. Thus another fiber from the same bundle was selected for the remainder of the experiment and it is reported here. Note that this fiber had already been exposed to the first cycle. There is one outlier at $T=800^{\circ}\text{C}$ within second ramp up data in Fig.1. This outlier makes the sign of CTE negative. The data and CTE without outlier are represented in Fig. 2. CTE without outlier has reasonable value and sign.

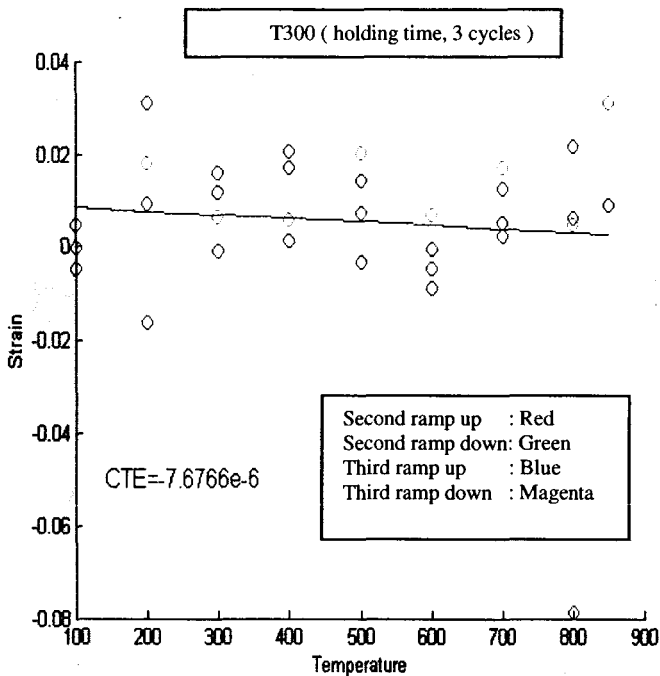


Fig. 1 CTE as obtained from the last two cycles with holding time.

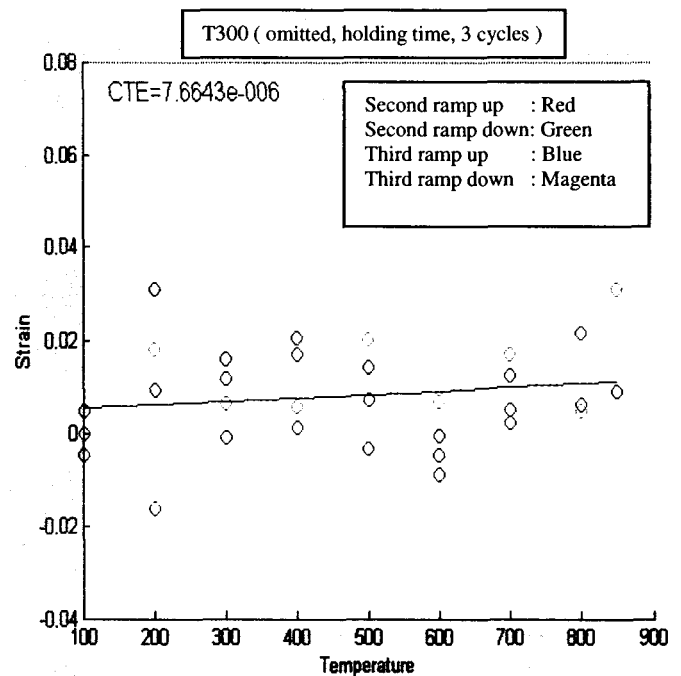


Fig. 2 CTE as obtained from the last two cycles with holding time excluding point at 800°C .

It takes 15 minutes to raise temperature up at increments of 100°C . The holding time at each constant temperature is 10 minutes. It takes 7 hours for just one cycle. Therefore, it is difficult to test for more than 4 cycles per day.

4. Future work

- Test K-1100 by holding time and 3 cycles and will analyze the data.
- Document surface roughness for T-300 by ESEM at room temperature.
- Start crystallinity observations for T-300 by x-ray spectroscopy.
- Statistical evaluation for the data from holding time, 2 cycles and 1 cycle, T-300.

List of fibers

Specimen	Treat	CO.	Piece
AS4			1(short)
T1000			1(long)
T900			1(long)
T650			1(long)
T300			1(long)
P55			1(long)
IM8			1(long)
IM7			1(long)
M30S-TORAY			1(long)
INCO			a little
FOAM		AFRL	3~4
T300	Heat treated after rigidization	BF Goodrich aerospace	6~7
T300	Heat treated after PYC	BF Goodrich aerospace	4~5
T300	Pan based	AMOCO	1 box
T650	Pan based	AMOCO	1 box
P55J2K	Pitch based	AMOCO	1 box
ULTMS-G,3K		HEXCEL	1 bundle
CVI Matrix		Rockwell science center	1 Piece
SLURRY Matrix		Rockwell science center	1 Piece
T300		Rockwell science center	3 bundles
T300	Infiltrated with AHPSCS polymer→SiC (5/11)	Rockwell science center	1 bundle

List of papers

1. M. Trinque, J.L. Carlier, A. Derre, P. Delhaes and P. Chadeyron, "High Temperature Thermal and Mechanical Properties of High Tensile Carbon Single Filaments," Carbon , Vol. 34, 1996, pp.923-929.
2. Hill, R., "Theory of Mechanical Properties of Fibre-Strengthened Materials-I. Elastic Behavior," J. of the Mechanics and Physics of Solids, Vol. 12, 1964, pp. 199-21
3. C. M. Neubauer and H. M. Jennings, "The Role of the environmental Scanning Electron Microscope in the Investigation of Cement-Based Materials," Scanning, Vol. 18, 1996, 515-521.
4. L. M. Jenkins and A. M. Donald, "Use of the environmental Scanning Electron Microscope for the Observation of the Swelling Behavior of Cellulosic Fibres," Scanning, Vol. 19, 1997, 92-97.

THERMOPHYSICAL ESEM & TEM CHARACTERIZATION of CARBON FIBERS

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Monthly Progress Report

June 2001

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Accomplishments

1. Monthly Summary

Week of	
June 4	<ul style="list-style-type: none">• Prepared EDS (Energy-Dispersive X-ray Spectrometry) and Surface roughness Test.
June 11	<ul style="list-style-type: none">• EDS and Surface roughness Test.• Used WDS (Wavelength-Dispersive X-ray Spectrometry) to do Multi-element quantitative analysis of fibers in unpolished section.• Prepared specimen with polished section for WDS Test.
June 18	<ul style="list-style-type: none">• Prepared specimen with polished section for WDS Test.• Completed WDS Test.• Completed EDS and Surface roughness Test.
June 25	<ul style="list-style-type: none">• Arranged the data and pictures from EDS, Surface roughness and WDS Tests• Attempted ESEM Test (holding time, 3 cycles) for NASA-T1000

2. Focus Areas

- a) Tested all specimens using EDS to know existence of Nitrogen.
- b) Tested NASA-T1000, NASA-T650, NASA-T300, NASA-IM7, BFG-T300-HT-RIDIG, BFG-T300-HT-PYC, AMOCO-P55J2K using WDS to quantitate Nitrogen.
- c) Used SEM that has better resolution than ESEM at room temperature to research surface roughness for all specimens.
- d) Tried to test NASA-T1000 using ESEM to achieve CTE by holding time, 3cycles test.

3. Results

a) EDS Test

The result of EDS Test shows that all fibers have only carbon except AMOCO-P55J2K that contains sulfur. It is very difficult for EDS to detect very small amount of element like Nitrogen. And EDS is usually used for qualitative analysis.

b) WDS test

Following table is The result of WDS Test. NASA-1000, NASA T650, NASA T300, NASA IM7 fibers have more Nitrogen than BFG-T300-HT-RIGID and -PYC and AMOCO-P552K. Specially, AMOCO-P55J2K fiber has not Nitrogen.

Specimen name	NASA-T1000	NASA-T650	NASA-T300	NASA-IM7	BFG-T300-HT-RIGID	BFG-T301-HT-PYC	AMOCO-P55J2K
Specimen Num.	2	4	5	8	12	13	16
First(%)	3.81	4.55	5.46	3.68	2.67	2.63	0.32
Second(%)	4.88	4.23	5.46	3.93	2.51	2.93	0.00
Third(%)	3.54	4.61	6.10	4.36	2.43	2.12	0.00
Fourth(%)	4.06	4.20	5.45	4.11	2.94	2.83	0.07
Average(%)	4.07	4.39	5.62	4.02	2.64	2.63	0.10

Table 1. Weight percentage of Nitrogen in each fiber by WDS.

c) Surface roughness.

Following 3 pictures were taken pictures using SEM at 9000X. Each fiber has different diameter. NASA T900 has very smooth surface. Fig 2. shows that BFG-T300-HT-RIGID is coated. The surface of NASA-T300 fiber has lines according to fiber longitudinal direction.

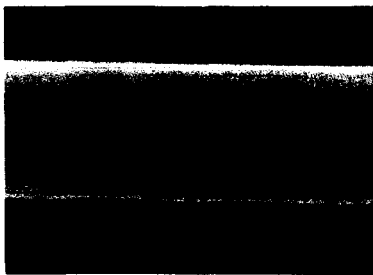


Fig.1 NASA T900

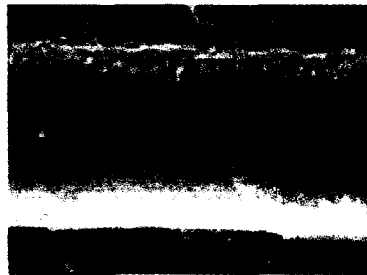


Fig.2 BFG-T300-HT-RIGID

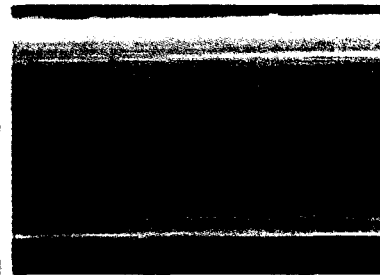


Fig.3 NASA-T300

1. Fiber that has coat: BFG-T300-HT-RIGID, BFG-T300-HT-PYC
2. Fiber that is smooth: HEXCEL-UHMS-G, NASA-AS4, NASA-IM7, NASA-IM8, NASA-M30S-TORAY, NASA-T1000, NASA-T900
3. Fiber that has line: AMOCO-P55J2K, AMOCO-T300, AMOCO-T650, NASA-T300, NASA-T650, RSC-T300

4. Future work

- a) Test NASA-T1000, AMOCO-P55J2K by holding time and 3 cycles and will analyze the data.
- b) Start crystallinity observations for T-300 by x-ray spectroscopy.
- c) Start FEA to analyze fiber behavior.

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Accomplishments

1. Monthly Summary

Week of	
July 2	<ul style="list-style-type: none"> • Took a rest because of infection.
July 9	<ul style="list-style-type: none"> • Attempted ESEM Test (with holding time, 3 cycles, NASA-T1000)
July 16	<ul style="list-style-type: none"> • Attempted ESEM Test (with holding time, 3 cycles, NASA-T1000) • Prepared specimen with polished section for WDS Test. • Looking for the method to reduce arching and overcharging for NASA-T1000 during ESEM thermal cycle Test.
July 23	<ul style="list-style-type: none"> • Prepared specimen with polished section for WDS Test. • Completed WDS Test. • Arranged the data and pictures of WDS Tests • Completed ESEM Test (holding time, 2 cycles) for NASA-T1000 • Analyzed the NASA-T1000 strain and CTE data and presented comparisons in tables and graphs

2. Focus Areas

- a) Tested NASA-T1000 (ESEM thermal cycle test), NASA-AS4, NASA-T900, NASA-IM8, NASA-P55, Hexcel UHMS-G using WDS to quantitate Nitrogen content.
- b) Completed the cyclic thermal testing of NASA-T1000 fiber in ESEM by holding the specimen at each temperature for 10 min. during ramp up and cool down (total of 2 cycles) and evaluated the corresponding strain and CTE.

3. Results

a) WDS test

Table 1 is The result of WDS Test. NASA-T1000(ESEM thermal cycle test), NASA-AS4, NASA-T900, NASA-IM8 have more Nitrogen than NASA-P55 and Hexcel UHMS-G,3k.

Specimen name	NASA-AS4	NASA-T1000 (2 Cycles ESEM Test)	NASA-T900	NASA-T300	NASA-P55	NASA-IM8	HEXCEL UHMS-G,3K
Specimen	1.00	2.00	3.00	5.00	6.00	7.00	17.00

Num.							
First(%)	4.80	4.14	4.33	4.44	0.00	3.53	0.80
Second(%)	5.35	3.86	3.80	4.79	0.22	4.02	0.52
Third(%)	5.43	3.93	4.22	4.72	0.00	3.97	0.97
Fourth(%)	5.37	4.36	4.34		0.01		0.12
Average(%)	5.24	4.07	4.17	4.65	0.06	3.84	0.60

Table 1. Weight percentage of Nitrogen in each fiber by WDS.

Table 2 is the weight percentage of Nitrogen on NASA-T1000 for which 2 Cycles ESEM test was not conducted and on one for which 2 Cycles ESEM test was conducted. Both result of NASA-T1000 and NASA-T1000(2 Cycles ESEM test) are almost same.

Specimen Name	First(%)	Second(%)	Third(%)	Fourth(%)	Average(%)
NASA-T1000	3.809	4.877	3.538	4.060	4.071
NASA-T1000 (2 Cycles ESEM test)	4.144	3.858	3.930	4.356	4.072

Table 2. Comparison of weight percentage of Nitrogen for NASA-T1000 and NASA-T1000(2 Cycles ESEM test)

b) Evaluation of circumferential CTE

Sometimes fibers were gone during ESEM thermal cycle test. Therefore, we got the image from two fibers at the same time during ESEM thermal cycle test. The result of

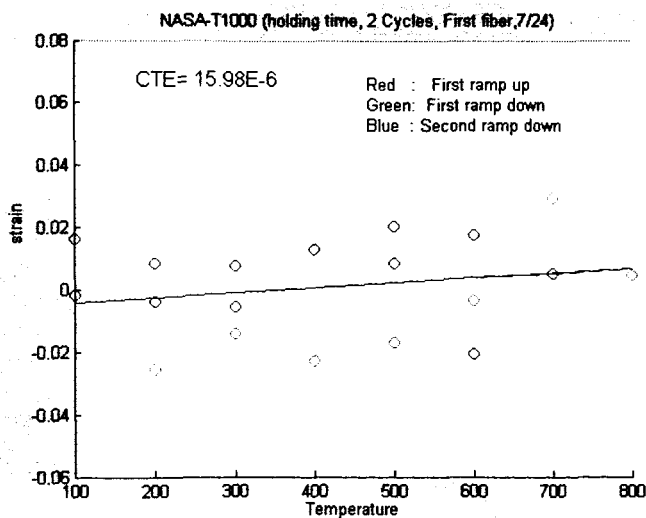


Fig. 1 CTE as obtained from first fiber.

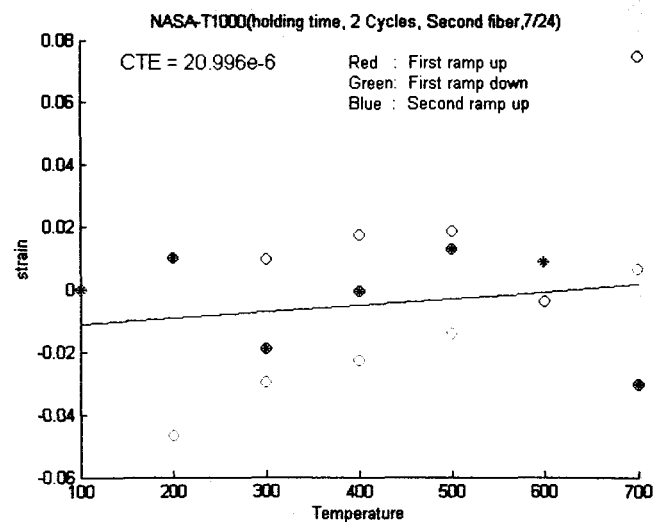


Fig. 2 CTE as obtained from second fiber

holding time test conducted over two cycles is presented in Figure 1 and Figure 2 where both ramp up and ramp down data are included. Thermal cyclic test using ESEM for NASA-T1000 had been attempted five times since June. However, at the high temperature ($T > 600$) much more arch and charging occurred for NASA-T1000 during ESEM thermal cycle test than for NASA-T300. It was very difficult to get a clear image of NASA-T1000 at high temperature.

4. Future work

- a) Test NASA-T1000, AMOCO-P55J2K by holding time and 3 cycles using new mounting method to reduce arch and overcharging during ESEM thermal cycle test and will analyze the data.
- b) Test crystallinity for T-300 by x-ray spectroscopy and TEM.
- c) Start FEA to analyze fiber behavior.

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Accomplishments

1. Monthly Summary

Week of	
August 6	<ul style="list-style-type: none"> Completed ESEM Test (3 fibers, holding time, 2 cycles) for AMOCO-P55J2K.
August 12	<ul style="list-style-type: none"> Completed ESEM Test (3 fibers, holding time, 3 cycles) for BFG-T300-HT-RIGID. Analyzed the AMOCO-P55J2K strain and CTE data and presented comparisons in graphs.
August 19	<ul style="list-style-type: none"> Analyzed BFG-T300-HT-RIGID strain and CTE data and presented comparisons in graphs. Conducted crystallinity test for NASA-T300 (Prior to ESEM test), NASA-T300 (Post ESEM cyclic test) and AMOCO-P55J2K by x-ray spectroscopy and TEM with Xhiping lu who is an expert for crystallinity at EM Center.
August 26	<ul style="list-style-type: none"> Conducted crystallinity test for BFG-T300-HT-RIGID (Prior to ESEM test) and BFG-T300-HT-RIGID (Post ESEM cyclic test) by x-ray spectroscopy and TEM. Analyzed crystallinity result for NASA-T300 (Prior to ESEM test), NASA-T300 (Post ESEM cyclic test), AMOCO-P55J2K, BFG-T300-HT-RIGID (Prior to ESEM test) and BFG-T300-HT-RIGID (Post ESEM cyclic test).

2. Focus Areas

- a) Completed the cyclic thermal testing of AMOCO-P55J2K and BFG-T300-HT-RIGID fiber in ESEM by holding the specimen at each temperature for 6 min. during ramp up and cool down (total of 3 cycles) and evaluated the corresponding strain and CTE.
- b) Obtained the crystallinity data with TEM of NASA-T300 (Prior to ESEM test), NASA-T300 (Post ESEM cyclic test) and AMOCO-P55J2K.

3. Results

3.1 AMOCO-P55J2K

3.1.1 Evaluation of circumferential CTE

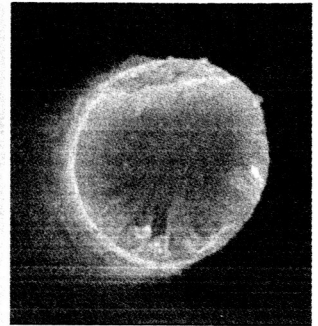
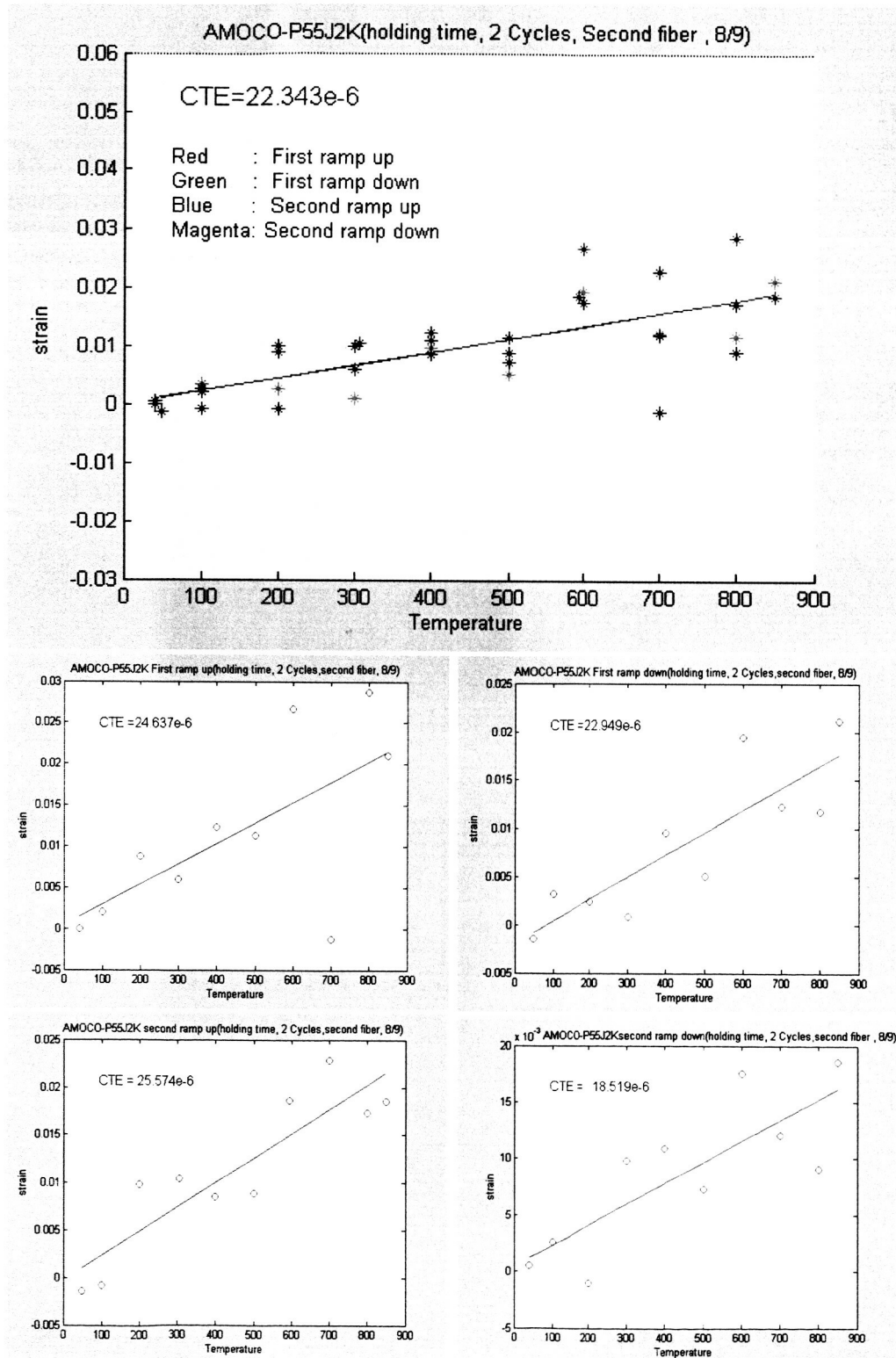


Fig 1. The CTE result for AMOCO-P55J2K Second fiber.

We got the images from three fibers at the same time by holding time test conducted over two cycles using ESEM Machine. The strain, CTE results for second fiber are presented in Figure 1 where both ramp up and ramp down data are included. The data from first and third fiber are in appendix. We consider only second fiber because the pictures of first and third fibers are ambiguous. The result shows that at the high temperature ($T > 600$), strain data is scattered.

3.1.2 TEM Results for AMOCO-P55J2K



Fig 2. HREM image for AMOCO-P55J2K

Fig. 2 is TEM image for AMOCO-P55J2K. There are nano-scale crystals that are Polycrystalline carbon-Graphite ($a=2.46 \text{ \AA}$, $c=6.711 \text{ \AA}$) and crystalline cover outer layers. Outer layer's crystalline has orientation ($[002]$) parallel to layer line.

3. 2 BFG-T300-HT-RIGID

3. 2.1 Evaluation of circumferential CTEs

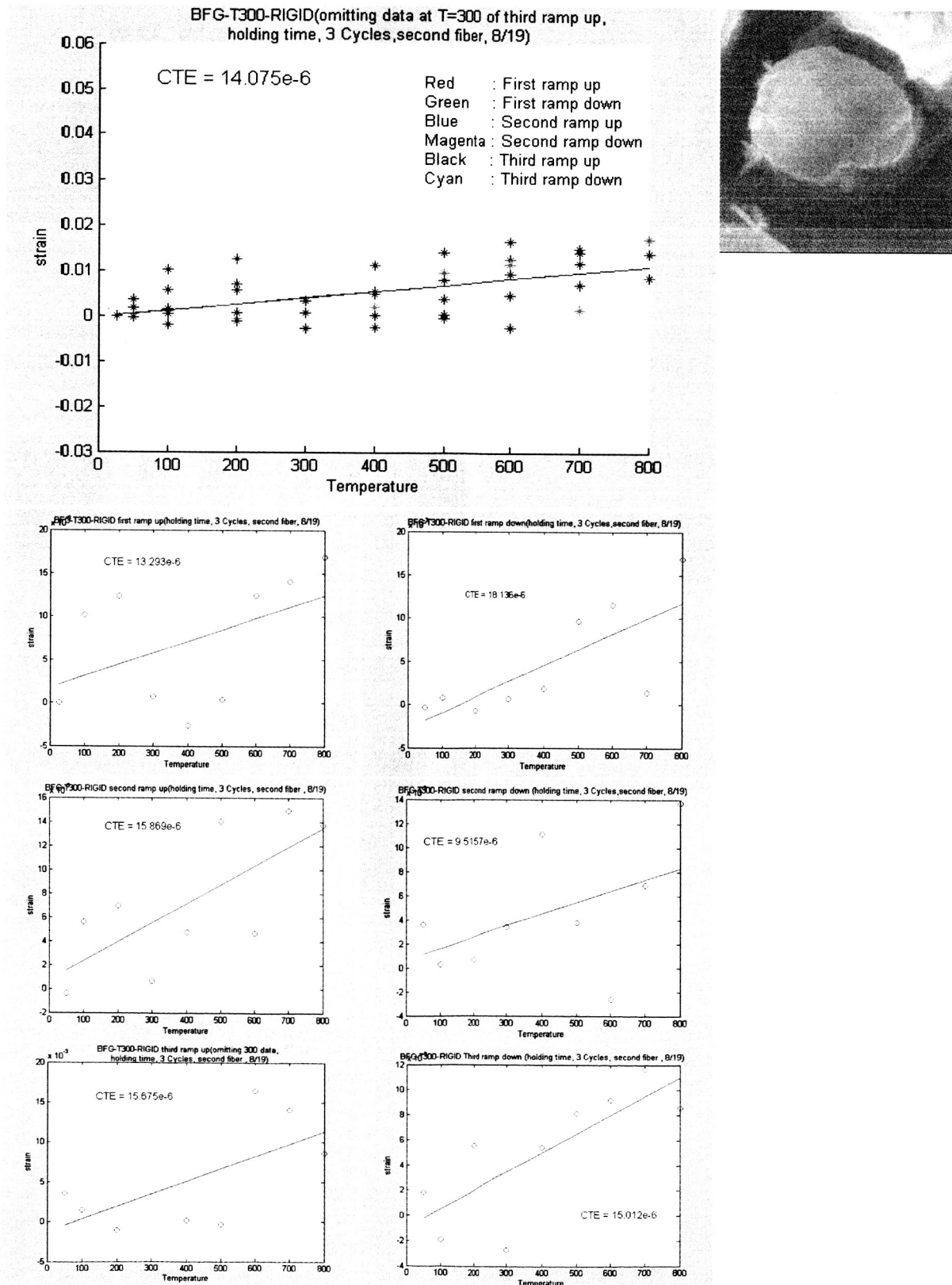


Fig 3. The CTE result for BFG-T300-HT-RIGID Second fiber.

We got the images from three fibers at the same time by holding time (6 min.) test conducted over three cycles using ESEM Machine. The strain, CTE results for second fiber are presented in Figure 3 where both ramp up and ramp down data are included. The data from first fiber are in appendix. We consider only second fiber because the pictures of first fiber are ambiguous. Similarly the third fiber pictures are also not very good. The pictures of third fiber are too ambiguous to calculate strain. Therefore, we ignore the data from third fiber. The values of strains at the room temperature are relatively closer to other values than those at the other temperatures. The values of CTE from ramp up cycles are consistent.

3.3 TEM Observation for NASA-T300

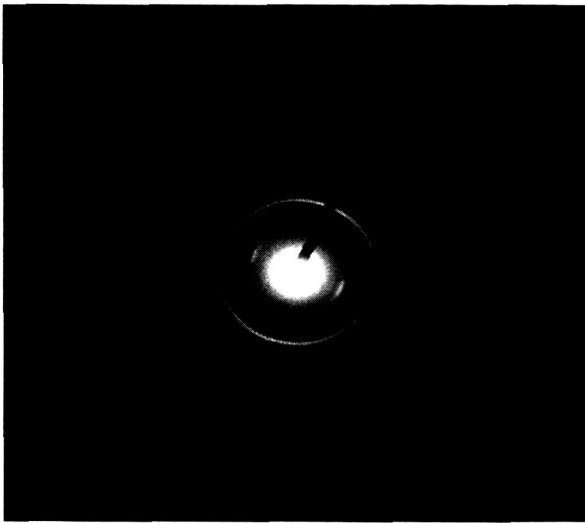


Fig 4. X-ray Diffraction Pattern for NASA-T300 (Prior to ESEM Test)

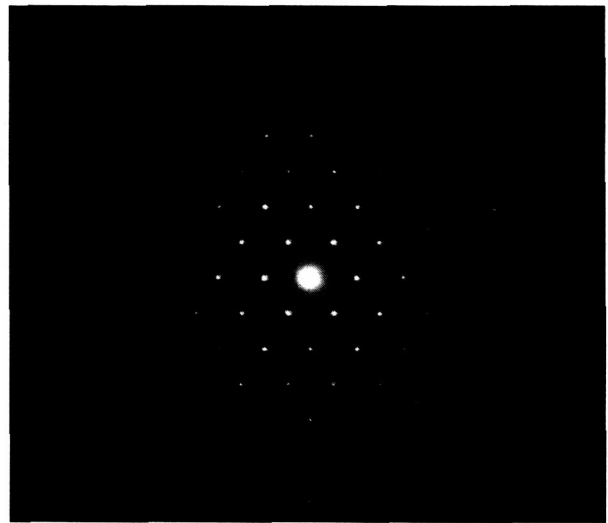


Fig 5. X-ray Diffraction Pattern for NASA-T300 (Post ESEM Cyclic test)

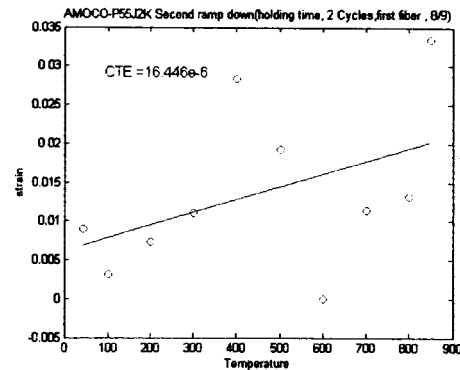
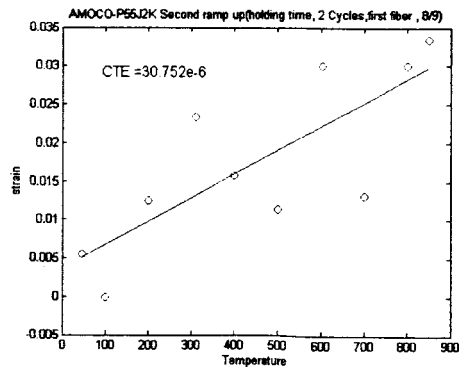
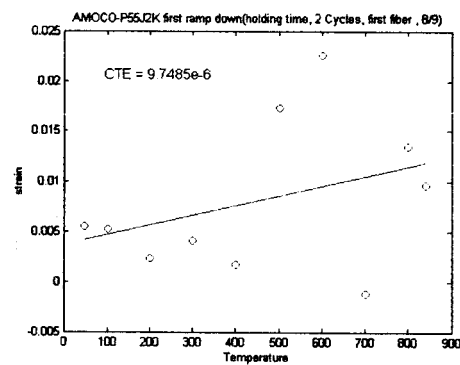
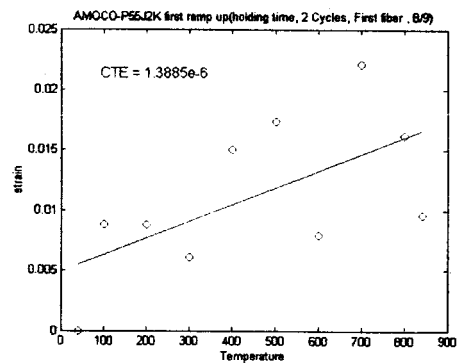
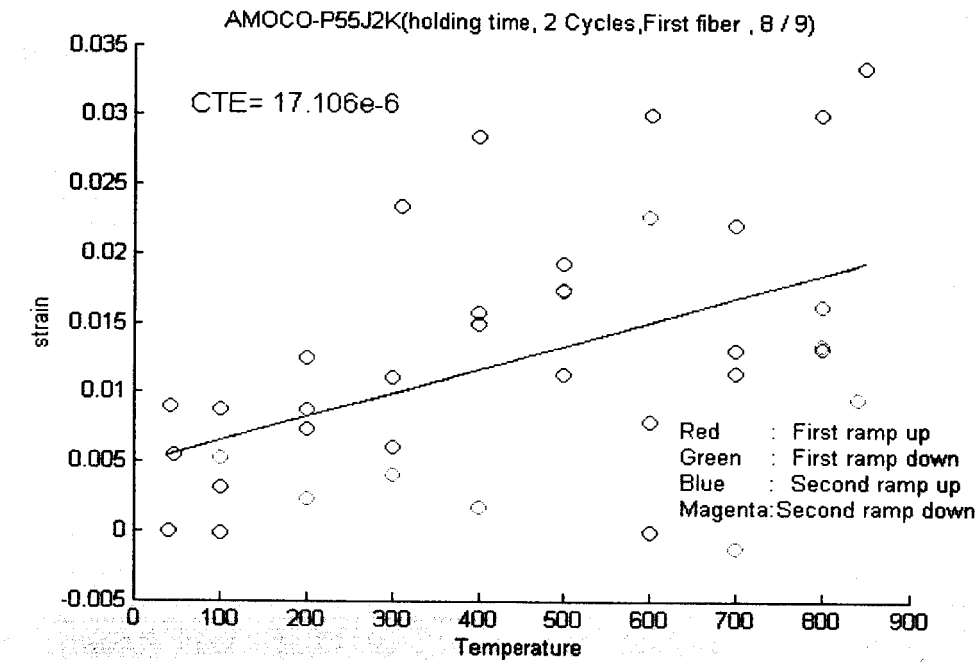
X-ray Diffraction Pattern image for NASA-T300 (Prior to ESEM Test) is presented in Fig. 4. It is composed of Polycrystalline carbon-graphite ($a=2.46 \text{ \AA}$, $c=6.711 \text{ \AA}$) and well textured with a common plane of [002]. X-ray Diffraction Pattern image for NASA-T300 (Post ESEM Cyclic test) is presented in Fig. 5, which is a different image than in Fig. 4. These changes indicate that now the crystalline particles grow more than one of NASA-T300 (Prior to ESEM Cyclic test) by ESEM Cyclic test. And there is the size change of crystal structure ($a=4.85 \text{ \AA}$, $c=5.2 \text{ \AA}$).

4. Future work

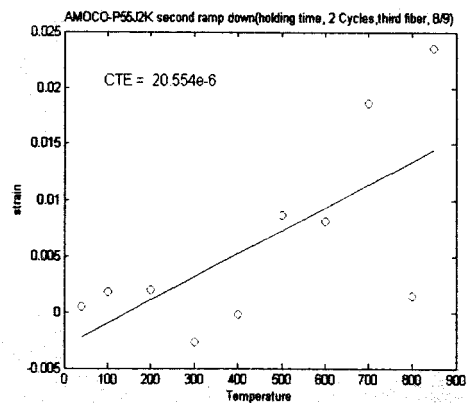
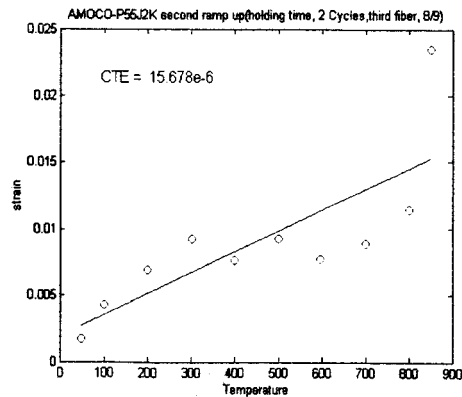
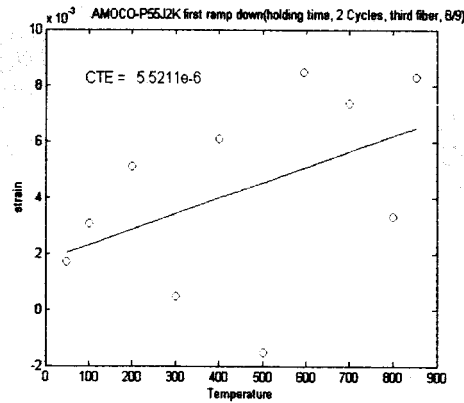
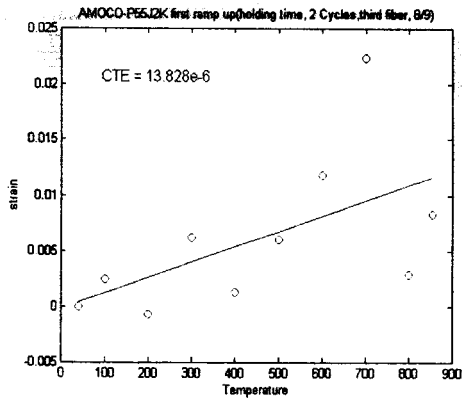
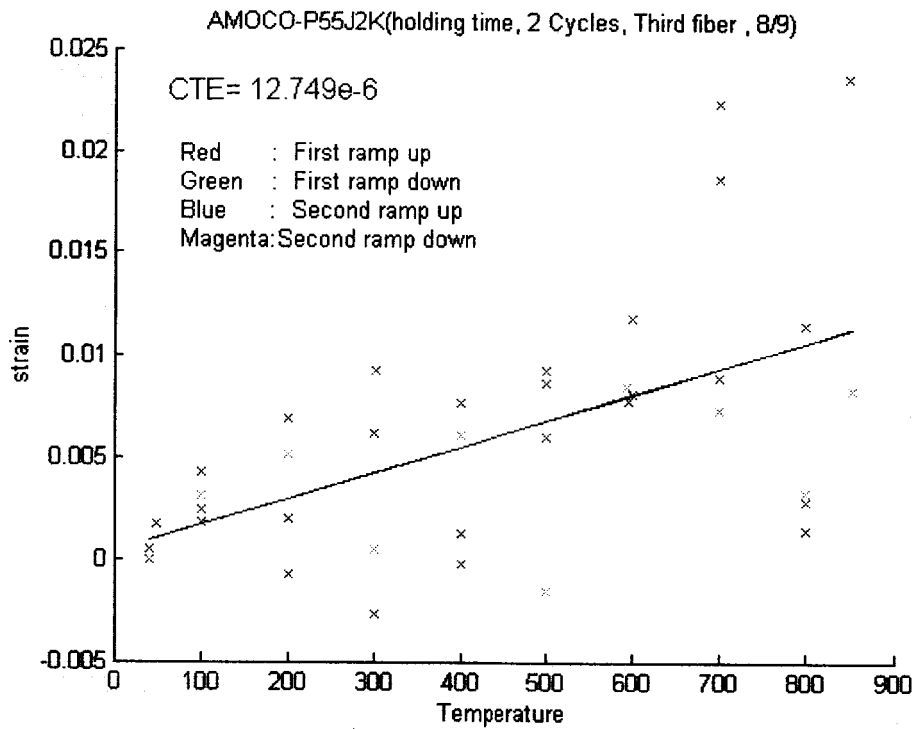
- a) Test crystallinity for BFG-T300-HT-RIGID (Prior to ESEM Test), BFG-T300-HT-RIGID (Post ESEM cyclic Test), BFG-T300-HT-PYC and NASA-T1000 by x-ray diffraction fringe and TEM.
- b) Focus on FEA to analyze CTE evaluation.
- c) Design the fixture for axial CTE measurement in ESEM.

* Appendix

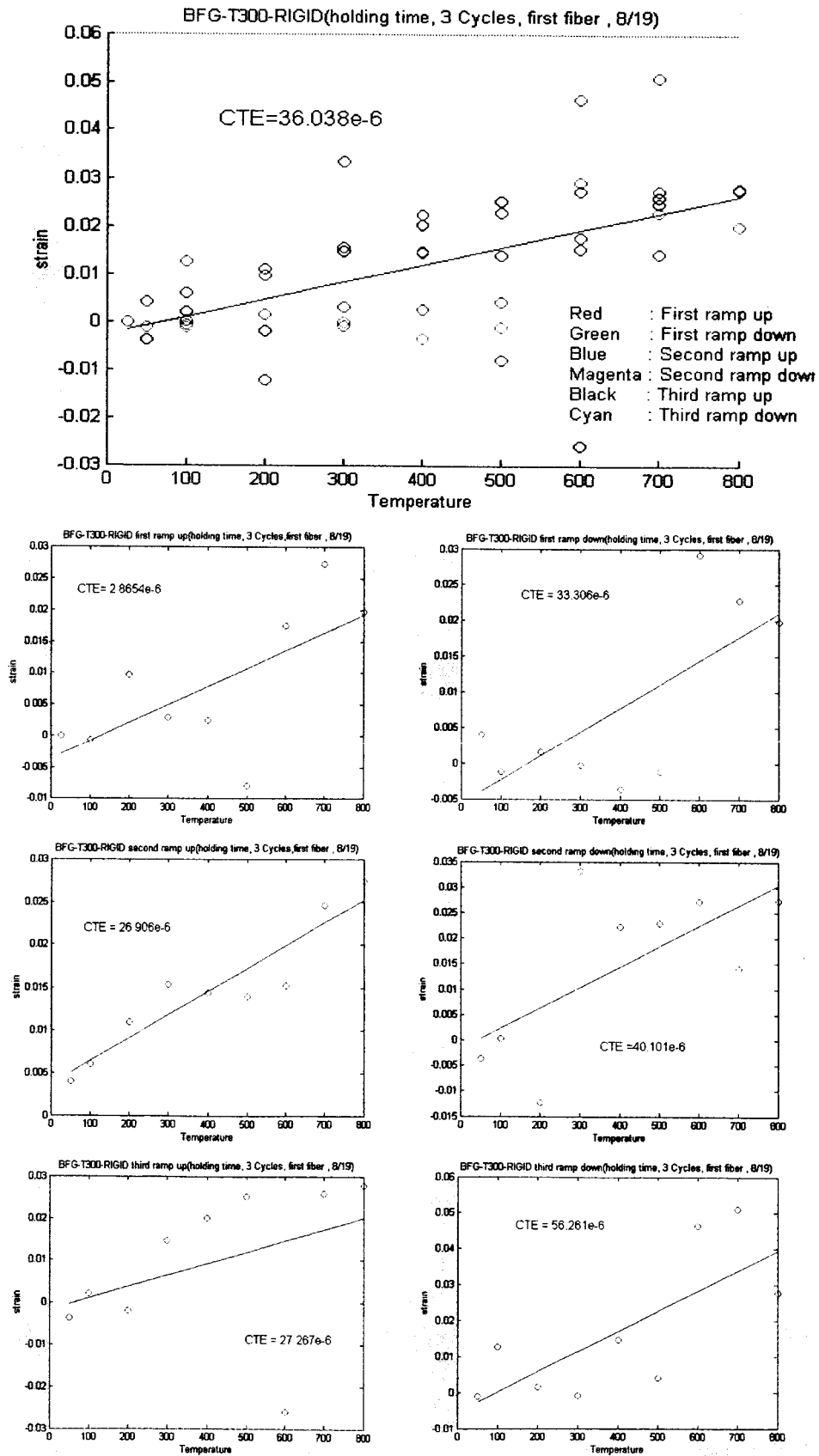
1. AMOCO-P55J2K fiber#1



2. AMOCO-P55J2K fiber #3



3. BFG-T300-HT-RIGID fiber #1



THERMOPHYSICAL ESEM & TEM CHARACTERIZATION of CARBON FIBERS

**CTE, SPECTROSCOPY and ROUGHNESS STUDIES at
HIGH TEMPERATURES
(Grant NAG 8 –1802)**

Monthly Progress Report

SEPTEMBER 2001

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Accomplishments

1. Monthly Summary

Week of	
September 3	<ul style="list-style-type: none"> • Process of finalizing the fixture for axial CTE measurement in ESEM. • Study Marc & Mentat (FEA program) to analyze CTE evaluation.
September 10	<ul style="list-style-type: none"> • Searched and read paper about crystallinity. • Combined and analyzed all data with relation to NASA-T300.
September 17	<ul style="list-style-type: none"> • Study Marc & Mentat (FEA program) to analyze CTE evaluation. • Combined and analyzed all data with relation to NASA-T1000.
September 24	<ul style="list-style-type: none"> • Combined and analyzed all data with relation to BFG-T300-RIGID. • Study Marc & Mentat (FEA program) to analyze CTE evaluation

2. Focus Areas

Combined and analyzed all data generated to date NASA-T300, NASA-T1000 and BFG-T300-RIGID fibers to find any potential correlations between TEM, XRD, SEM data in relation to the CTE values calculated.

3. Results

3.2 NASA-T300

3.2.1 *Evaluation of circumferential CTE*

TABLE 1. 1 THE LIST OF ESEM TEST FOR NASA-T300

	date		cycles
Test 1	10/17/2000	without holding time	1
Test 2	3/20/2001	without holding time	3
Test 3	4/5/2001	with holding time (10 min.)	1
Test 4	5/31/2001	with holding time(6 min.)	2

Fig. 1.1 CTE as obtained from ramp up data only (A type data set.)

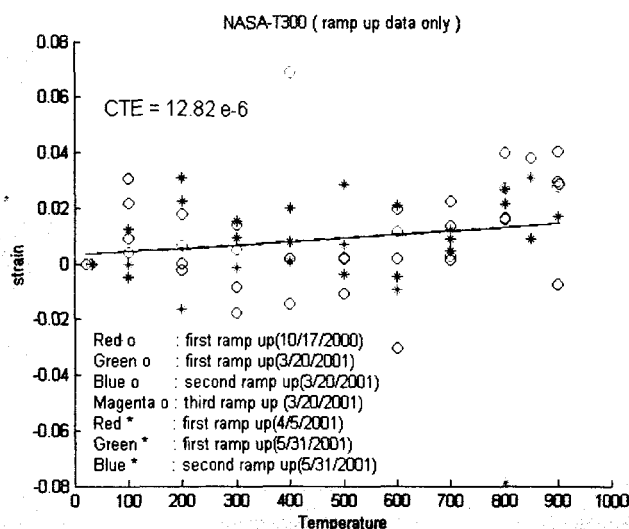


Fig. 1.2 CTE as obtained from ramp up and ramp down data in all tests. (C type data set.)

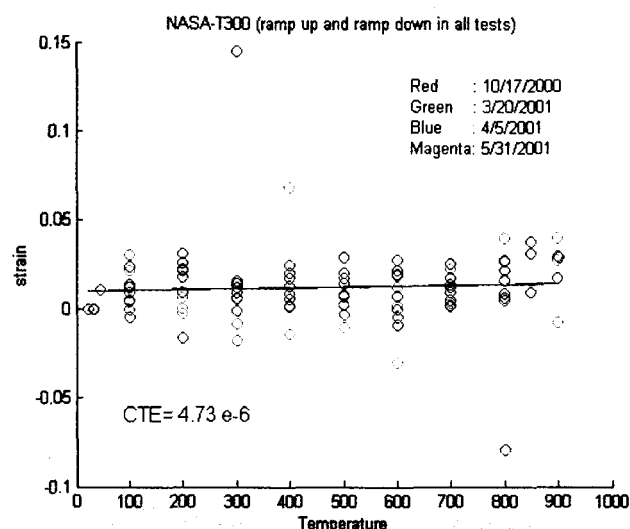


TABLE 1.2 THE LIST OF CTE CALCULATIONS FOR NASA-T300

NASA-T300	CTE (10^{-6})				
	A	B	C	D	E
Test 1	26.82	26.82	4.59	4.59	4.59
Test 2	15.92	18.56	15.93	18.56	15.93
Test 3	14.82	14.82	9.25	9.25	9.25
Test 4	-7.45	17.34	-7.68	7.66	7.66
*Total	12.82	19.71	4.72	13.37	12.72
A: Using ramp up data points only					
B: Using ramp up data and excluding data points corresponding to 400°C of Test #2 ramp up and 800°C of Test #4 ramp up					
C: Using ramp up and ramp down complete data set					
D: using ramp up and ramp down complete data set excluding data point corresponding to 300°C of Test #1 ramp down, 400°C of Test #2 ramp up and 800°C of Test #4 ramp up.					
E: using ramp up and ramp down complete data set excluding data point corresponding to 300°C of Test #1 ramp down and 800C of Test #4 ramp up.					

* Total: CTE for all data points of each column title.

Table 1.1 presents a list of ESEM tests for NASA-T300 fiber. In Test #4, we reduced holding time to 6 minutes per temperature increments in comparison to the holding time of Test #3 which was 10 minutes. Each single cycle whether ramp up or ramp up with 10 min hold takes about 7 hours to complete. Considering the size of the fiber and the heating chamber, we believe that 6 min hold is acceptable to reach stable heat transfer conditions.

Table 1.2 shows that CTE for *Total data using ramp up data points only(A) is much different from one for *Total data using ramp up and ramp down complete data set(C). CTE values of *Total data for A, D and E are similar to each other. The CTE results of *Total data for D and E came from *Total data for C excluding outlier data points. Therefore, we can say that if we have enough data, CTE will approach the value between 12×10^{-6} and 13×10^{-6} . Certainly more insight is needed to address the fine differences among the ramp up vs ramp down vs all inclusive [total] calculations.

3.1.2. WDS, TEM for X-ray diffraction pattern and surface roughness test

- Weight percentage of Nitrogen in each fiber by WDS (Wavelength-Dispersive X-ray Spectrometry) is 5.62% for NASA-T300. This percentage value is highest one among fibers that we have. We will continue to pay attention to any corroboration between CTE and Weight percentage of Nitrogen.
- TEM test to obtain the X-ray diffraction pattern for Test #4

Prior to ESEM Cyclic Test, polycrystalline carbon-graphite size is $a=2.46 \text{ \AA}$, $c=6.711 \text{ \AA}$

Post ESEM Cyclic test, polycrystalline carbon-graphite size is $a=4.85 \text{ \AA}$, $c=5.2 \text{ \AA}$. This indicates that there is some size change in the crystal structure. Figures 1. 3 and Fig 1.4 clearly illustrate this change as well.

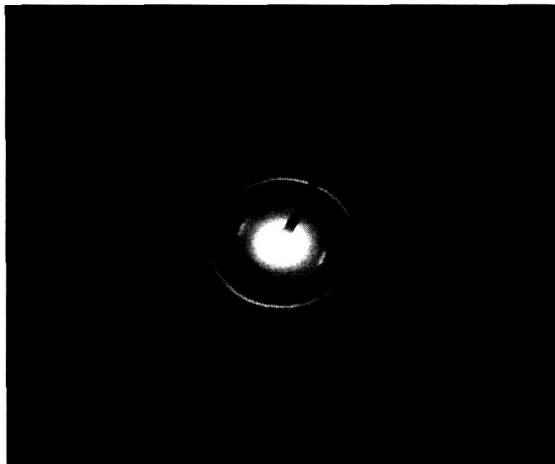


Fig 1.3 X-ray Diffraction Pattern for NASA-T300 (Prior to ESEM)

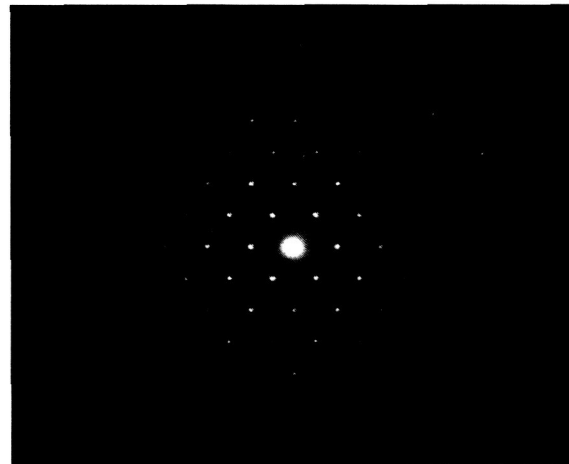


Fig 1. 4 X-ray Diffraction Pattern for NASA -T300 (Post ESEM)

- The surface roughness of this fiber is captured in the SEM image of Fig 1.5. This picture was taken at 9000X magnification. Note that the diameter in this image is 5.09 micrometer. The ridges(lines) make the perimeter measurements somewhat difficult when the cross

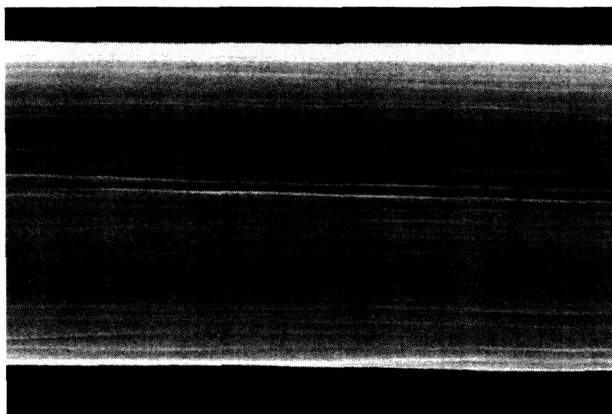


Fig 1.5 SEM image of NASA-T300 fiber

section is viewed during the thermal cycling ESEM tests

3. 2 NASA-T1000

3. 2.1 Evaluation of circumferential CTEs

TABLE 2. 1 THE LIST OF ESEM TESTS FOR NASA-T1000

	date		cycles
Test 1	10/3/2000	without holding time	3
Test 2	1/16/2001	without holding time	2
Test 3 *	7/24/2001 *	with holding time(6 Min.)	2

*Test 3: 2 fibers are used simultaneously for ESEM Cyclic test.

TABLE 2. 2 THE LIST OF CTE RESULT FOR NASA-T1000

	CTE (10^{-6})	
NASA-T000	A	B
Test 1	26.32	26.32
Test 2	7.75	6.82
Test 3	-7.16	17.62
*Total	8.89	15.24
A: Using ramp up data points only		
B: Using ramp up and ramp down complete data set		

* Total: CTE for all data point of each column title.

At first site, the data presented below may appear to be more scattered than those presented in Fig 1.1-1.2. This is only due to the different scales. Note that for NASA-T1000 all strain data points are between -0.04 and 0.04.

Fig. 2. 1 CTE as obtained from ramp up
(A type data set.)

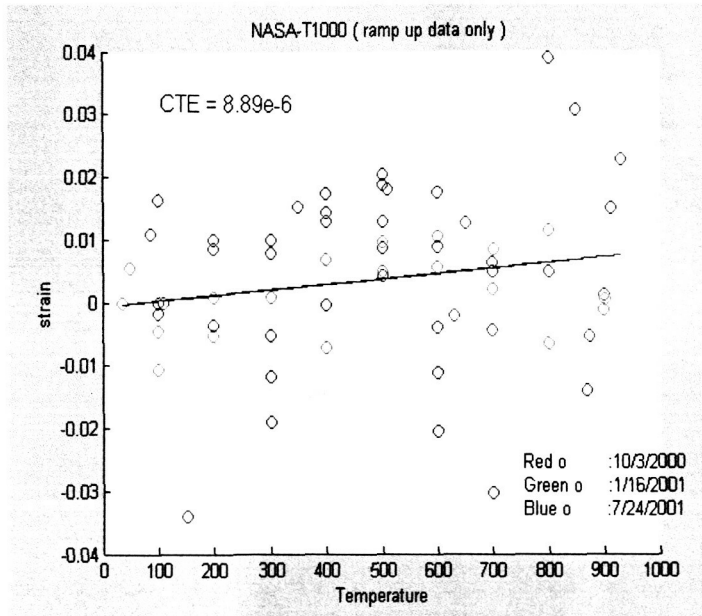
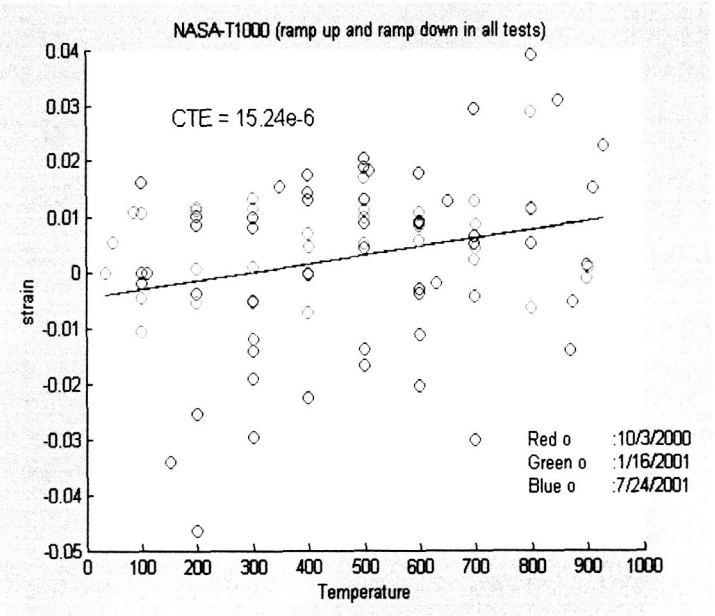


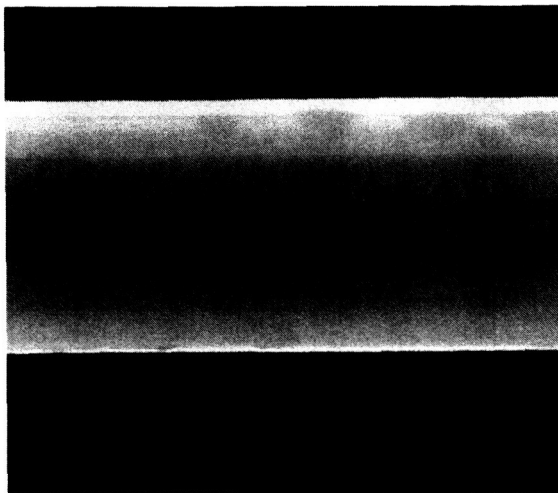
Fig. 2.2 CTE as obtained from ramp up and ramp
down data in all tests. (B type data set.)



3.2.2. WDS and surface roughness test

- Weight percentage of Nitrogen for NASA-T1000 by WDS (Wavelength-Dispersive X-ray Spectrometry) is 4.07% for NASA-T1000.
- TEM test to get X-ray diffraction pattern has not been completed.
- The surface roughness at 9000X is presented in Fig 2.3. The diameter in this image is 5.09 micrometer. As seen, the NASA-T1000 fiber is smooth. We believe that this feature also impacts the accuracy of the perimeter calculations; in this case we content that these results are more accurate.

Fig 2. 3 SEM image for NASA-T1000



3.3 BFG T300-RIGID

3.3.1 Evaluation of circumferential CTEs

TABLE 3. 1 THE LIST OF ESEM TEST FOR BFG-300

	Date		Cycles
*Test 1	8/19/2001	With holding time (6 min.)	2

*Test 1: 2 fibers are used simultaneously for ESEM Cyclic test.

TABLE 3. 2 THE LIST OF CTE RESULT FOR BFG-300

	CTE(10^{-6})	
BFG-T300	A	B
Test 1	18.46	24.15
A: Using ramp up data points only		
B: Using ramp up and ramp down complete data set		

As can be observed in Table 3.2, CTE values for BFG-T300-RIGID is much higher than those of other fibers. However we can not confirm the above situation since we are in the process of conducting additional tests. In the near future, we can compare CTE values with more confidence.

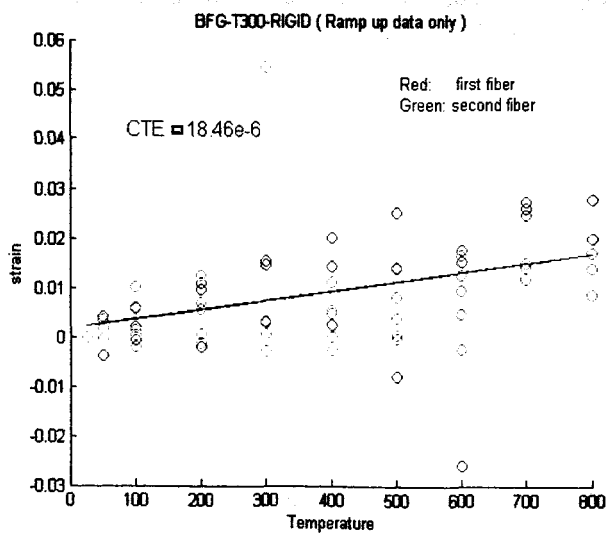


Fig. 3. 1 CTE as obtained from ramp up
(A type data set.)

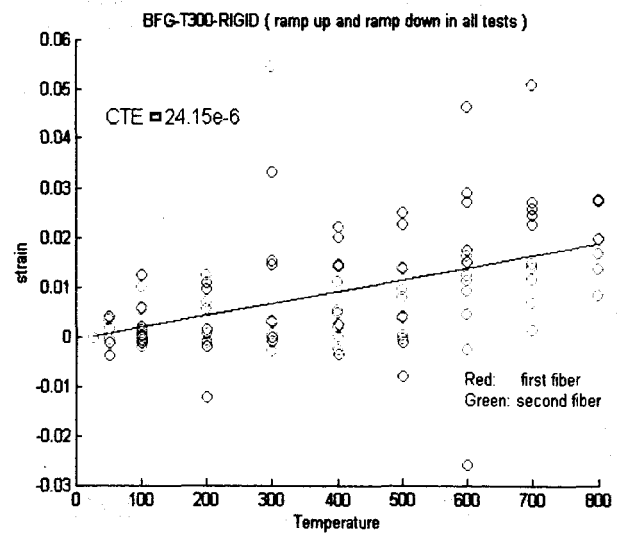


Fig. 3.2 CTE as obtained from ramp up and
ramp down data in all tests. (B type data set.)

3. 3. 2. WDS, TEM for X-ray diffraction pattern and surface roughness test

- Weight percentage of Nitrogen for BFG-T300-RIGID by WDS is 2.64%. This value is comparably small among the other fibers that we have. Can we say that the smaller is weight percentage of Nitrogen in fiber, the higher value of CTE is? We need more data...
- TEM test shows that polycrystalline carbon-graphite size is $a=4.85 \text{ \AA}$, $c=5.2 \text{ \AA}$. This polycrystalline carbon-graphite size is identical to the NASA-T300 fiber for post ESEM Cyclic test. But we expect that different percentage of size distribution may have taken place. Now, we are looking for a method to measure this percentage.
- Fig 3.3 and Fig 3.4 were taken at 9000X magnitude. The diameter from Fig 3.3 image is 6.9 micrometer and the diameter from Fig 3. 4 image is 8.03 micrometer. BFG-T300-RIGID fiber definitely has a coat. Fig 3.3 shows that fiber's coat has peeled off at some sites whereas in Fig 3.4, coat is not damaged.

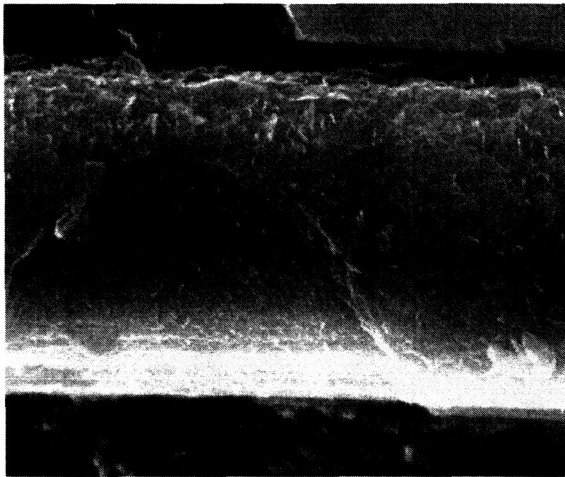


Fig 3. 3 SEM image for BFG-T300-RIGID

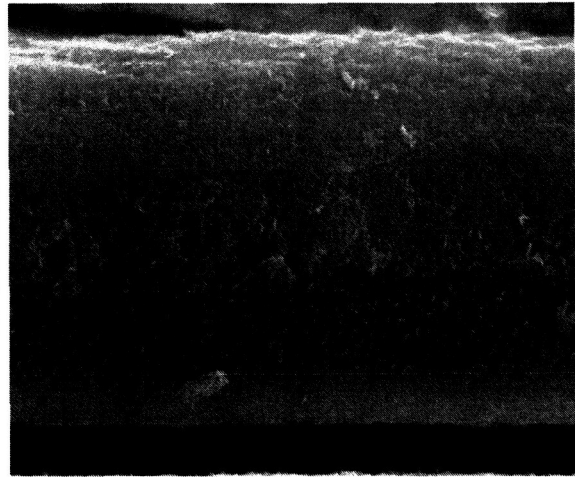


Fig 3. 4 SEM image for BFG-T300-RIGID

4. Future work

- a) TEM test to get X-ray diffraction pattern for NASA-T1000, BFG-T300-PYC and Hexcel-UHMS-G,3K by x-ray diffraction fringe and TEM.
- b) Finish designing the fixture for axial CTE measurement in ESEM.
- c) Test NASA-T1000, AMOCO-P55J2K by holding time and 3 cycles and will analyze the data.
- d) Model FEA to analyze fiber behavior

List of papers

- a) M. Guigon, A. Oberlin and G. Desarmot, "Microtexture and Structure of some High Tensile Strength, PAN-Base Carbon Fibres," *Fibre Science and Technology* v. 20 (1984) 55-72.
- b) R. J. Diefendorf and E. Tokarsky, "High-Performance Carbon Fibers", *Polymer Engineering and Science*, v.15 (1975) 150-159.
- c) B. J. Wicks and F. A. Coyle, "Microstructural inhomogeneity in carbon fibres", *J. Materials Science* v. 11 (1976) 376-383.
- d) S. C. Bennett and D. J. Johnson, "Electron-Microscope Studies of Structural Heterogeneity in PAN-Based Carbon Fibres" *Carbon* v. 17 (1978) 25-39.

THERMOPHYSICAL ESEM & TEM CHARACTERIZATION of CARBON FIBERS

**CTE, SPECTROSCOPY and ROUGHNESS STUDIES at
HIGH TEMPERATURES
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Monthly Progress Report

OCTOBER 2001

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Accomplishments

1. Monthly Summary

Week of	
October 8	<ul style="list-style-type: none"> Created FEA model of the fiber subjected to isothermal temperature
October 15	<ul style="list-style-type: none"> TEM test to get X-ray diffraction pattern for NASA-T1000, BFG-T300-PYC and Hexcel-UHMS-G, 3K by x-ray diffraction fringe and TEM. FEA Model
October 22	<ul style="list-style-type: none"> Completed ESEM Test (3 fibers, holding time (6 min.), 2 cycles) for NASA-T1000 Analyzed the NASA-T1000 strain and CTE data
October 28	<ul style="list-style-type: none"> Completed another ESEM Test (2 fibers, holding time (6 min.), 3 cycles) for NASA-T1000 Analyzed the NASA-T1000 strain and CTE data Combined all strain data to date with relation to NASA-T1000.

2. Focus Areas

- a) FEM model to analyze fiber thermal behavior.
- b) Completed two ESEM tests for NASA-T1000 fiber; combined and analyzed all strain data generated to date on NASA-T1000 fibers
- c) Obtained TEM data for

NASA-T1000 (post ESEM test 10/ 24/ 2001)

NASA-T1000 (post ESEM test 10/ 29/ 2001)

NASA-T1000 (as received).

Hexcel-UHMS-G (as received)

BFG-T300-PYC (as received)

BFG-T300-Rigid (as received)

3. Results

3.3 NASA-T1000

3.3.1 ESEM test result for 10/ 24/ 2001 test

We took images from three fibers at the same time. The test was performed with holding time of 6 min. over two cycles. The strain, and corresponding CTE values for NASA-T1000 fiber tested on 10 / 24 / 2001 are presented in Figure 1.1 where only the ramp up data sets are included. As shown, the CTE results from first ramp up data are scattered, but the second ramp up data is considerably consistent with each other. Therefore, there is a possibility that after first ramp up, the fiber may have stabilized in its movement.

3.1.2. ESEM test result for 10/ 29/ 2001 test

The strain, CTE results for NASA-T1000 fiber tested at 10 / 29 / 2001 are presented in Figure 1.2 where only ramp up data sets are included. We took the images from two fibers at the same time. The test was performed with a holding time (6 min.) over three cycles. The clarity of the images was poorer in general then those of pictures from 10/ 24/ 2001 ESEM test. Thus we do not consider this set as a viable set.

3.1.3. All data sets comparison

Test date	CTE(e-6) from each data set	CTE(e-6) from accumulated data sets *
10/3/2000	26.320	26.32
1/16/2001	8.722	7.753
7/24/2001	-7.169	8.891
10/24/2001	13.454	7.753
10/29/2001	6.660	7.759

Table 1 CTE Result for NASA-T1000 (Ramp up data only).

The CTE results for each of the NASA-T1000 ESEM Tests are presented in Table 1. First column is the name of the data set which reflects the test date. Second column reports the CTE result corresponding to the test date. In the third column, we use all data sets available on that date. When we consider CTE values of the second column, we are not able to find any trends. However, the cumulative calculations presented in the third column converge rapidly. This trend is also shown in Fig. 1.4. We will pursue this further by examining closely the ramp up values and the image quality issues in each test.

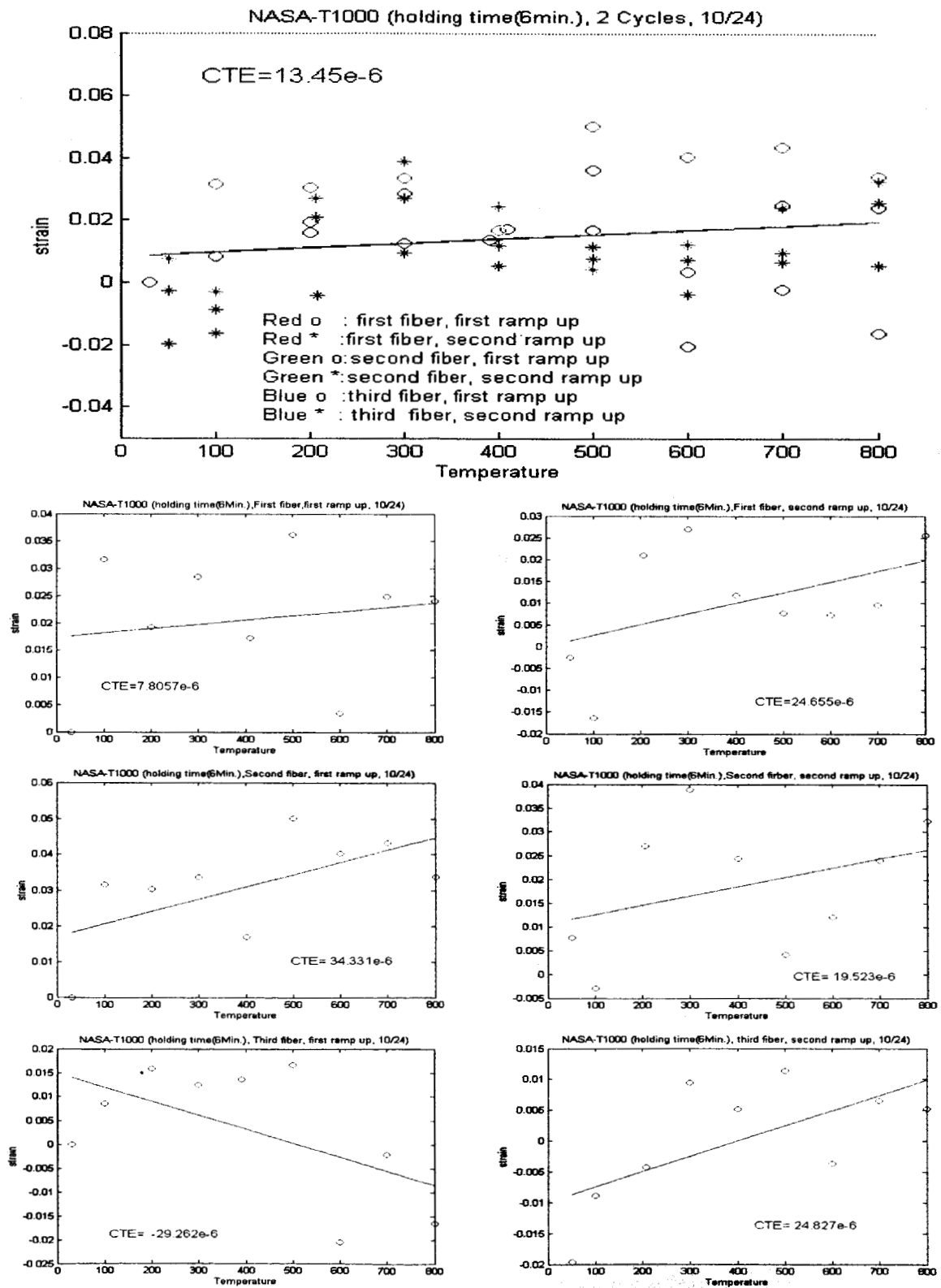


Fig 1.1 ESEM Test result for NASA-T1000 (10 / 24 / 2001 test)

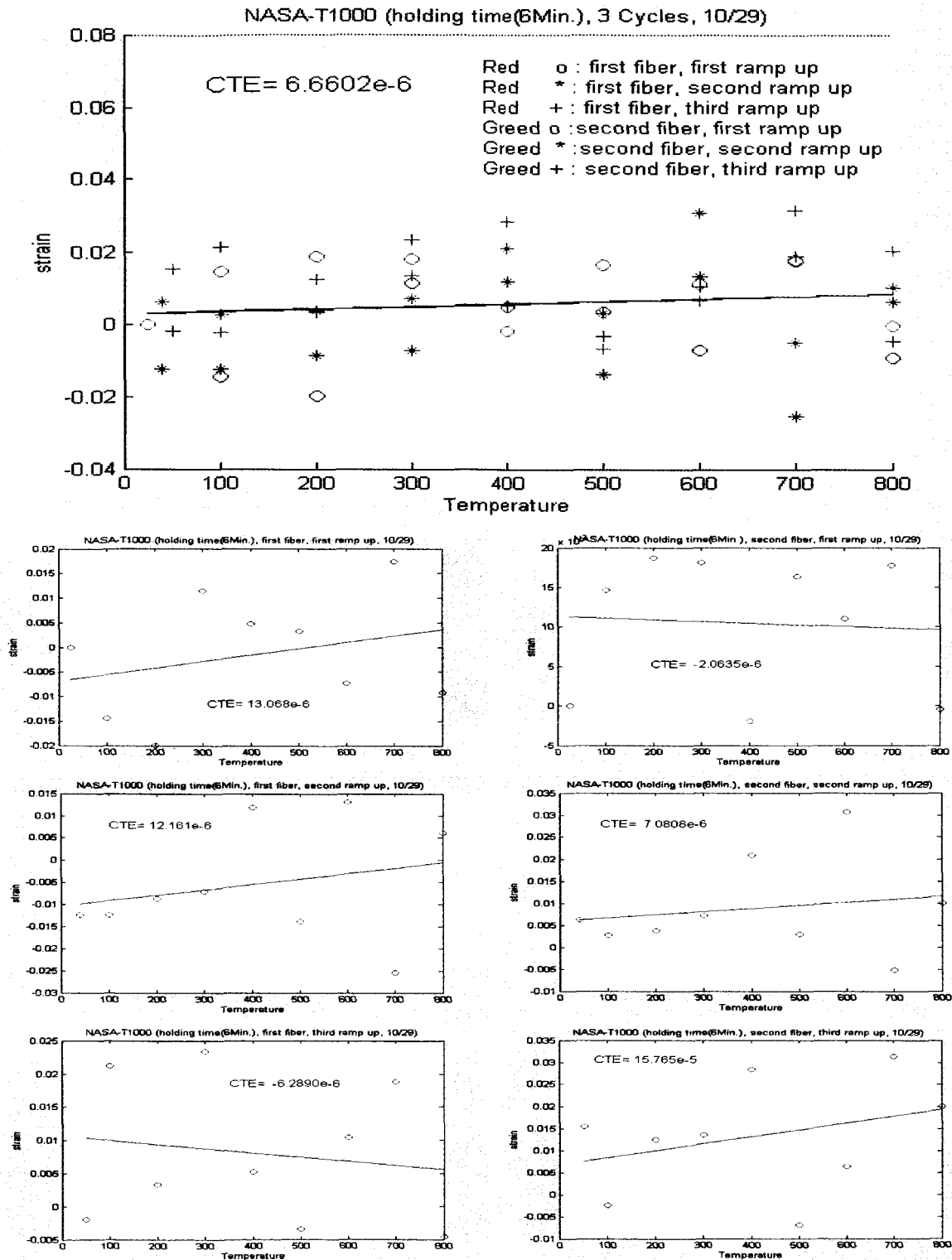


Fig 1.2 ESEM Test result for NASA-T1000 (10 / 29 / 2001 test)

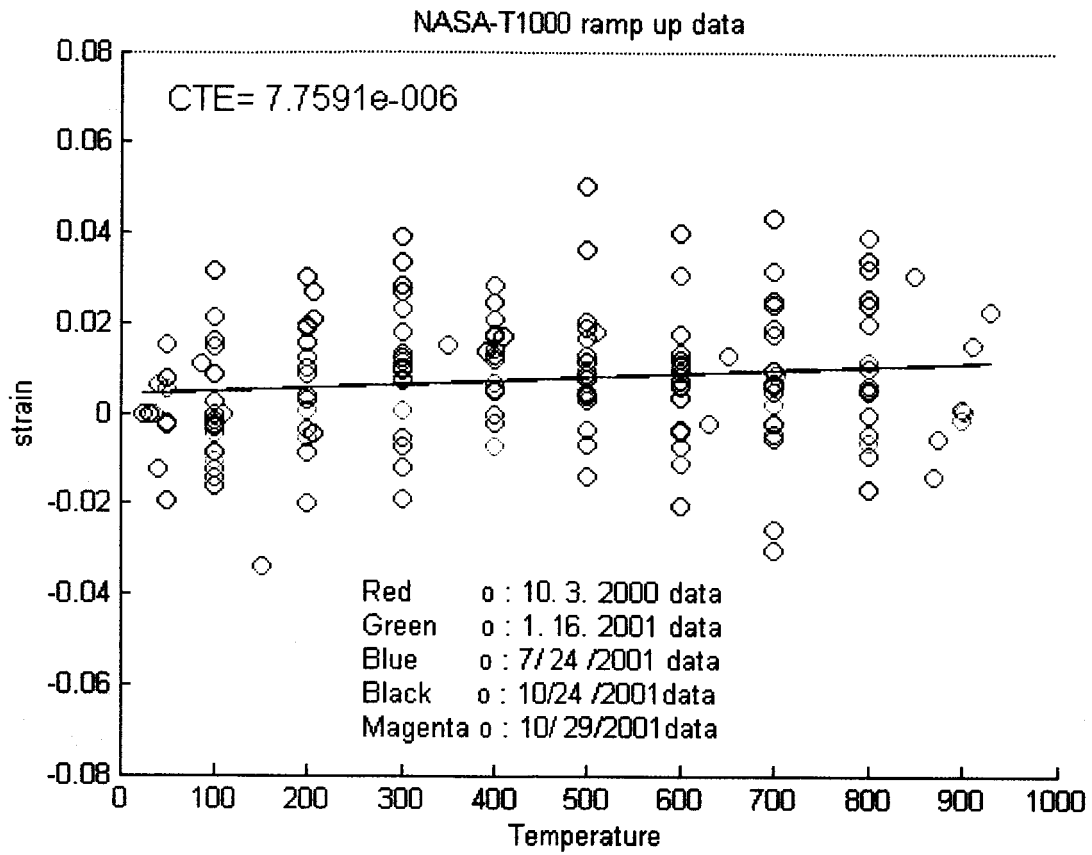


Fig. 1.3 Strains as obtained from ramp up of all test sets for NASA-T1000 fiber

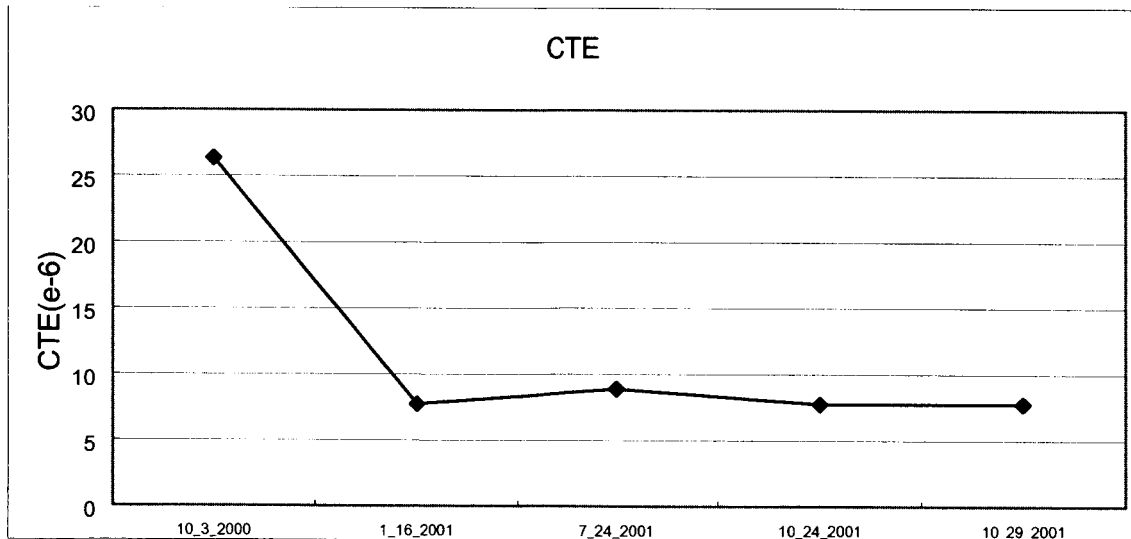


Fig 1.4 CTE values for accumulated data set

3. 2 FEA Model

A simple FEA model for the fibers (Diameter: 5.09 micro-meter) with ANSYS is completed. In this two dimensional plane strain model, Plane 42 element type which has two degrees of freedom (U_x , U_y) is used. In this time, isotropic material behavior ($E_x=255$ GPa, Density= 1770 Kg/m^3 , $G_{xy}=23.1$ GPa, CTE= $7.759\text{E-}6$) is considered and temperature is applied as a body force (from 20 C to 800 C). Deformed and undeformed shapes are presented in Fig. 3.1. Dashed line is undeformed shape (original) and solid line is deformed shape. This result had constant thermal strain across the cross section and matched the theoretical value.

Thermal strain from FEA Model: $e_x=0.6062\text{e-}2$, $e_y=0.6062\text{e-}2$, $e_z=0.6062\text{e-}2$

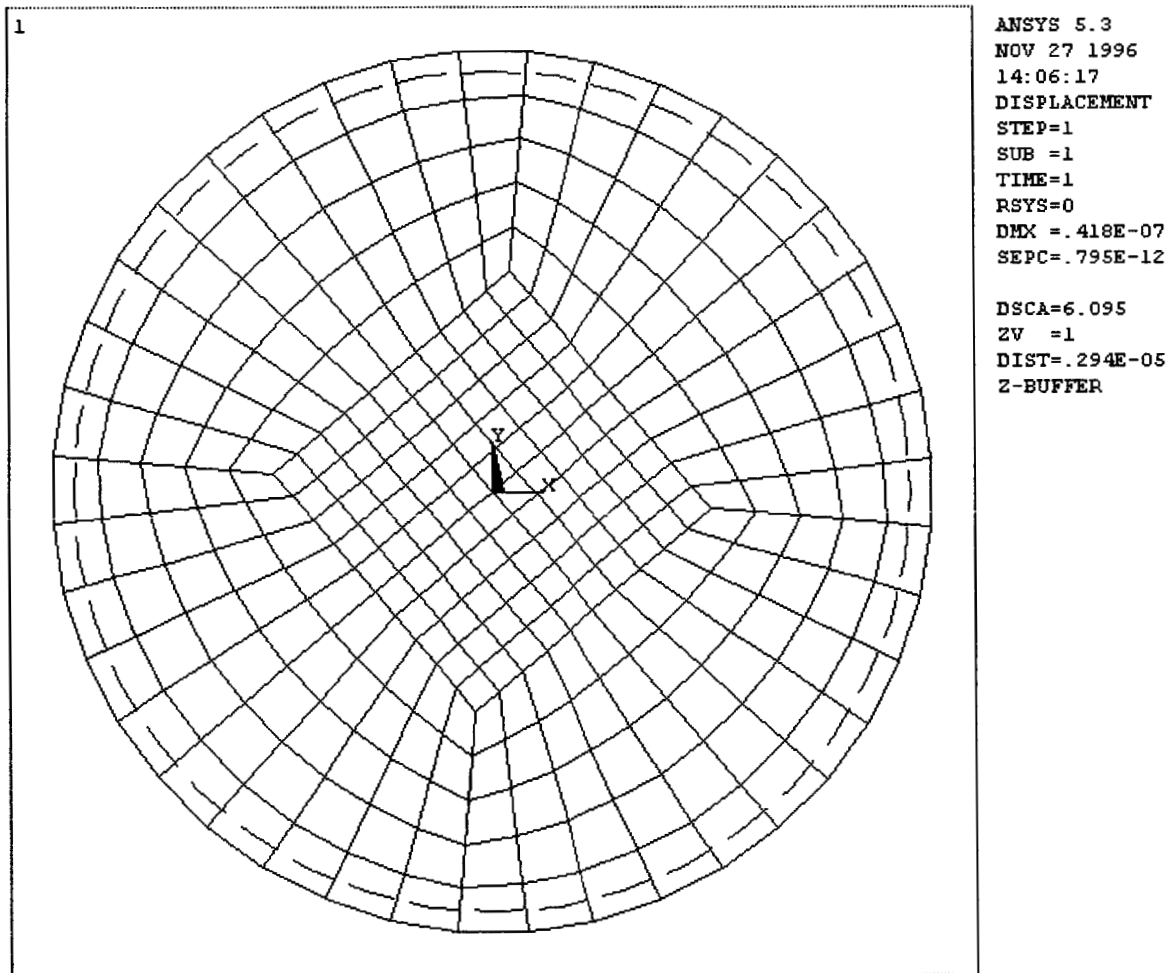


Fig. 3.1 Deformed and undeformed displacements of a single fiber

3.4 TEM Results

The electron diffraction patterns were obtained for the following fibers; NASA-T1000 (post test 10/ 24/ 2001), NASA-T1000 (post test 10/ 29/ 2001), Hexcel-UHMS-G (as received), BFG-T300-PYC (as received), BFG-T300-Rigid (as received) and NASA-T1000 (as received). All fibers displayed the same graphite structure (polycrystalline carbon-graphite size : $a=2.46$, $c=6.711$). JEOL 2010 system was used for NASA-T1000 (post test 10/ 24/ 2001) and NASA-T1000 (post test 10/ 29/ 2001) and Zeiss10C was used for Hexcel-UHMS -G (as received), BFG-T300-PYC (as received), BFG-T300-Rigid (as received) and NASA-T1000 (as received) since JEOL 2010 was out of order at the time (now it is repaired).

The result for NASA-T1000 (post test 10/ 24/ 2001) is presented in Fig. 3.3. Note that the fiber longitudinal axis is perpendicular to the orientation of C axis.

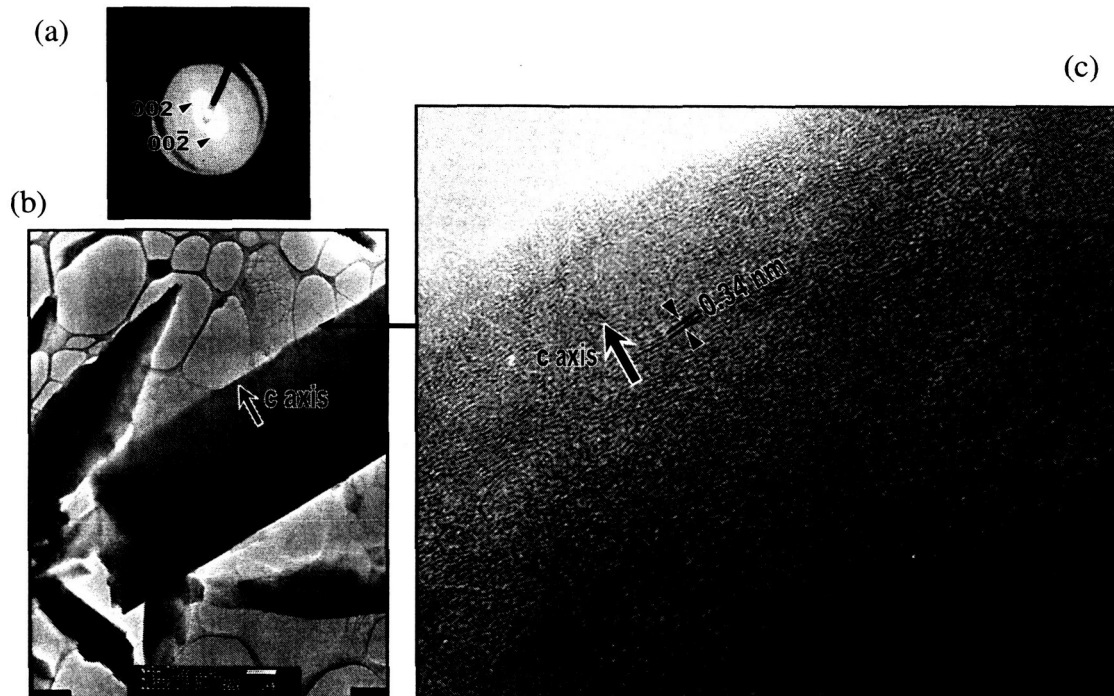


Fig. 3.3 Tem result for NASA-T1000 (10/24/2001)

- (a) Electron diffraction pattern
- (b) Segment of crushed fiber (X6000)
- (c) C spacing at X3200,000

4. Future work

- a) Repeat tests for electron diffraction pattern of NASA-T1000, NASA-T300 using the new system by which we can check the position of fiber cross-section
- b) Finish the fixture for axial CTE measurement in ESEM.
- c) Re-evaluate mounting of the fiber to assure positional stability in the ESEM
- d) Model FEA to analyze fiber behavior using orthotropic material properties in polar coordinates.

THERMOPHYSICAL ESEM & TEM CHARACTERIZATION of CARBON FIBERS

**CTE, SPECTROSCOPY and ROUGHNESS STUDIES at
HIGH TEMPERATURES
(Grant NAG 8 –1802)**

Monthly Progress Report

NOVEMBER 2001

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Accomplishments

1. Monthly Summary

Week of	
November 5	<ul style="list-style-type: none">• FEM model to estimate transverse CTE using optimization method.• Searching for mounting method of the fiber to assure positional stability in the ESEM
November 12	<ul style="list-style-type: none">• Searching for mounting method of the fiber to assure positional stability in the ESEM
November 19	<ul style="list-style-type: none">• Electron diffraction pattern of NASA-T1000 using the new system by which we can check the position of fiber cross-section.• Searching for mounting method of the fiber to assure positional stability in the ESEM
November 26	<ul style="list-style-type: none">• FEM model to estimate transverse CTE using optimization method.• Test the new method (fiber-slicing method) to assure positional stability in the ESEM

2. Focus Areas

- a) Mounting method of the fiber to assure positional stability in the ESEM
- b) FEM model to estimate transverse CTE using optimization method.
- c) Electron diffraction pattern of NASA-T1000 using the new system by which we can check the position of fiber cross-section.

3. Results

3.4 New method to assure positional stability in the ESEM

New method was attempted to assure positional stability in the ESEM.

-Procedure-

- Embed fibers into the epoxy and cure at room temperature.
- Cut fibers to 2 micrometers in length using a microtome.
- Put the fiber on the circular plate and heat the plate until epoxy evaporates.
- Then, place the plate with fibers only into ESEM machine. Do ESEM Test.

We refer to this method as Fiber Slicing method. NASA-T300 fibers were prepared with this method. When temperature was raised rapidly (from 22 C to 700 C during 10 min.), found that the fiber did not move and was very stable. There was no angular change for cross-section of fiber since the height of fiber is smaller than diameter in the case. We did not take pictures during this test. We only wanted to demonstrate to feasibility.

3.2 FEA Model for optimization method

A FEA model for the fibers was developed to calculate CTE through optimization method (first order method).

-Assumption-

- Transversely isotropic material. (Longitudinal and transverse direction)
- Cross-section is perfect circle. Therefore we can use diameter instead of perimeter.
- Plane strain.

-Optimization variable

- Objective Function = $\left| \text{Diameter from experiment} - \text{Diameter from FEA Model} \right|$
- Design Variable = CTE (transverse)

-Material property (x-y plane is cross-section of fiber)

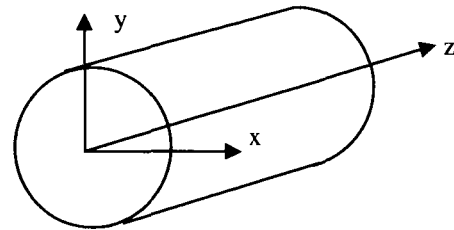
(From G. Wagoner, R. Bacon. "Elastic constants and Thermal Expansion Coefficients of Various Carbon Fibers." In extended abstracts of 19th Biennial carbon conference, Penn State University PP. 296-297, 1987)

$E_x=E_y=10.3 \text{ Gpa}$, $E_z=341 \text{ Gpa}$, $G_{xz}=G_{yz}=19.0\text{Gpa}$, $G_{xy}=3.5\text{Gpa}$, $\nu_{zx}=0.13$

CTE-xz=CTE-yz=-1.2 micro-meter

Fig. 1 FEA coordinate system

In this two dimensional plane strain models, Plane 42 element type of ANSYS package that has two degrees of freedom (U_x , U_y) is used and temperature is applied as a body force (from 20 C to 800 C).



-Procedure-

- Prepare FEA model to analyze fiber thermal behavior.
- Activate the optimization method (first order method, 20 iterations) and set tolerance
- Input experimental diameter at 800 C ($0.256975E-05$ in this time just choosing arbitrary value) and initial trial value ($6.0 E-6$) of design variable.
- Find design variable (CTE) for the minimum value of objective function.

In this example, the CTE value from optimization method is $7.7607E-6$ within 3.3% of value ($7.5E-6$). Objective function's dependence on iteration numbers is as shown below.

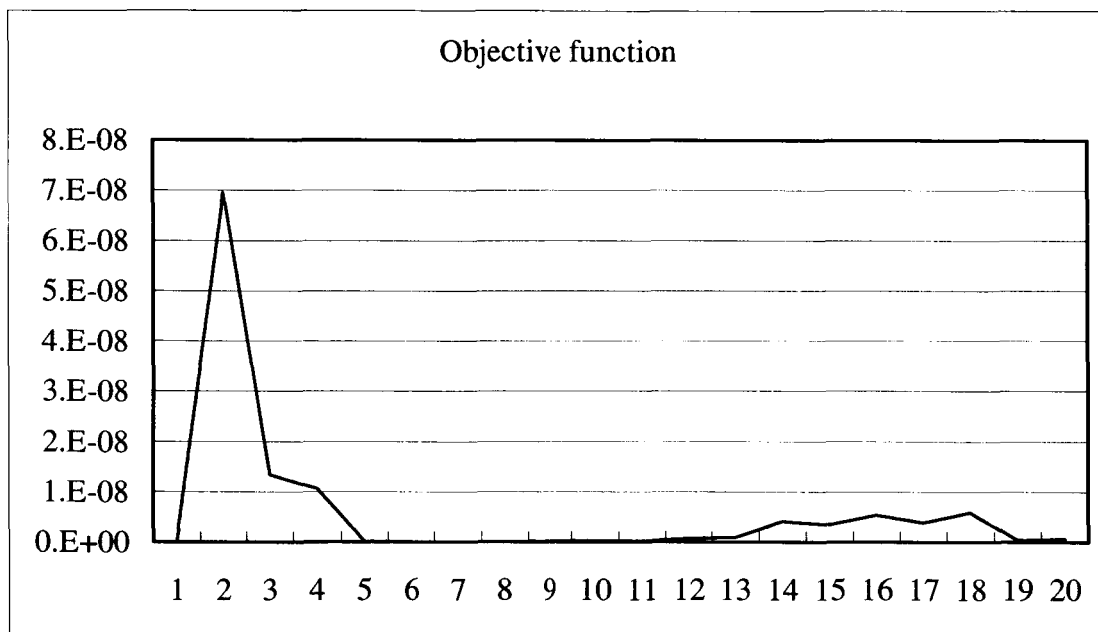


Fig. 2 Objective function

3.3 TEM Results

Electron diffraction patterns image were produced for NASA-T1000 ESEM-tested and as-received fibers using the new system by which we can check the position of fiber cross-section.

-Procedure-

1. Embed fibers into the epoxy and cure at room temperature.
2. Cut fibers by microtome using a diamond knife.
3. Place fibers into TEM machine. Produce Electron diffraction pattern using TEM picture.

TEM results are presented in Figure 3-4. Now, we can compare the EDP (Electron diffraction pattern) of core part with one of near surface. We can recognize the difference of EDP images from Fig. 3 and 4. EDP of core from Fig. 3 shows that structure is amorphous one. EDP of near surface from Fig. 3 shows that there is crystalline. When comparing Fig. 3 and Fig. 4, EDPs of near surface are different for each other. The difference shows that NASA-T1000 ESEM-tested has more crystalline than NASA-T1000 as-received. Therefore, ESEM heat cycle test make an effect on the structure of near surface.

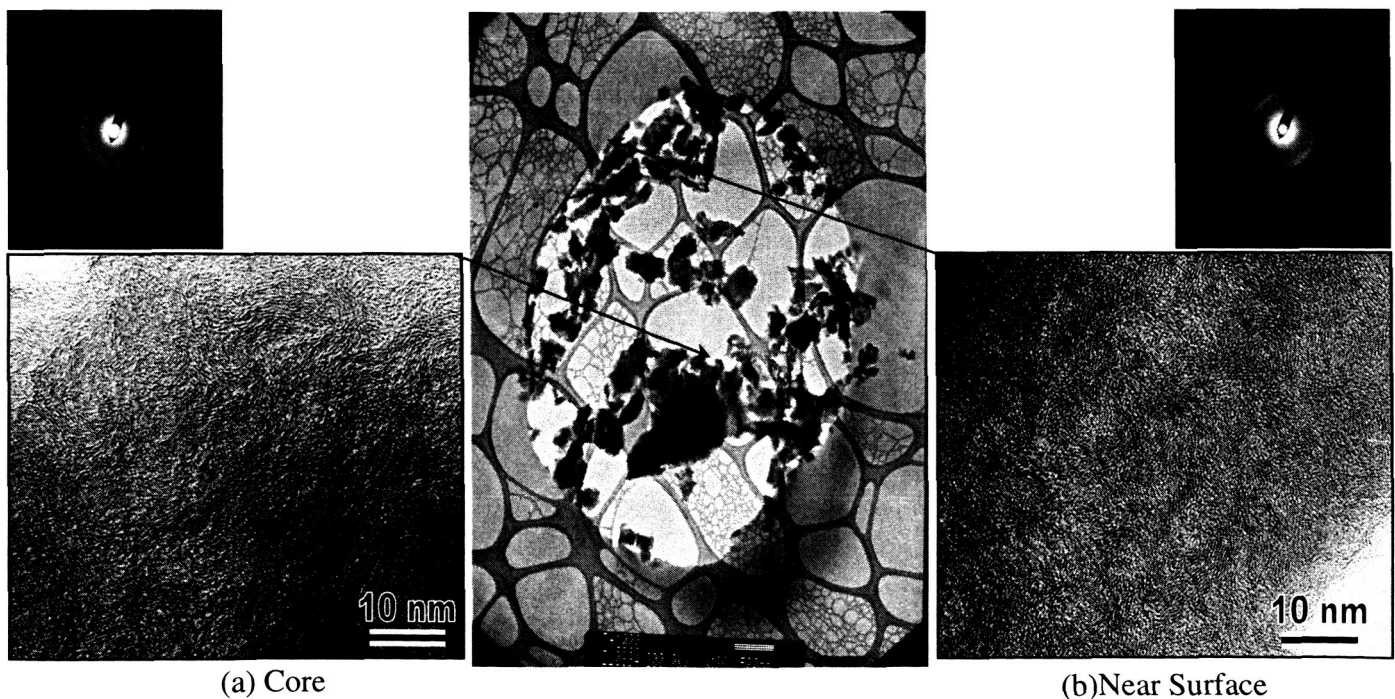


Fig.3 T-1000 as-received

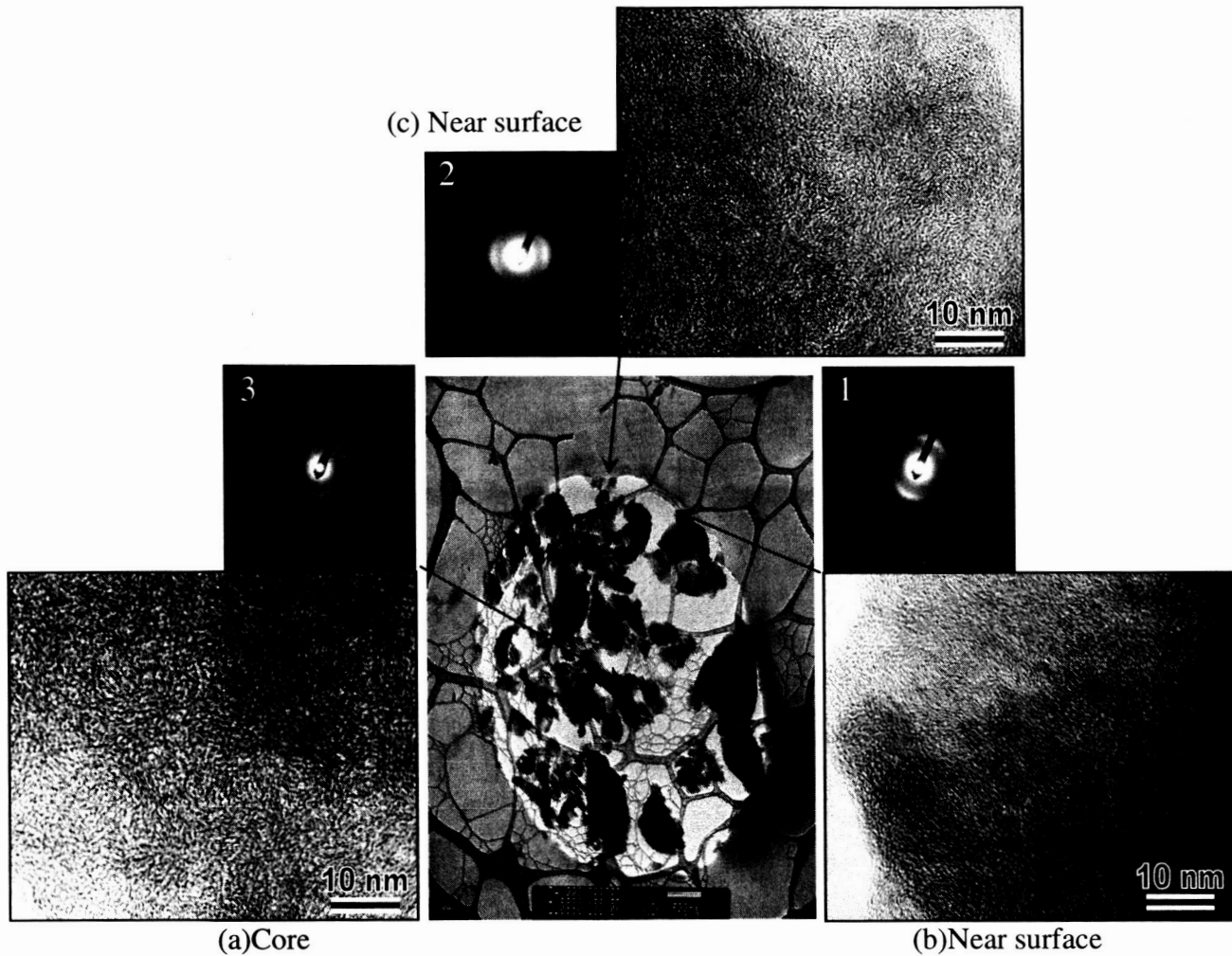


Fig 4. T-1000 ESEM-Tested

4. Future work

- TEM tests for electron diffraction pattern of NASA-T300, BFG-T300-Rigid and BFG-T300-PYC using the new system by which we can check the position of fiber cross-section.
- ESEM Test for NASA-T300 and NASA-T1000 fiber using fiber-slicing method to assure repeatability.
- 3D-Model FEA to estimate CTE of fiber using optimization method.

THERMOPHYSICAL ESEM & TEM CHARACTERIZATION of CARBON FIBERS

**CTE, SPECTROSCOPY and ROUGHNESS STUDIES at
HIGH TEMPERATURES
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Monthly Progress Report

January 2002

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Accomplishments

1. Monthly Summary

Week of	
January 7	<ul style="list-style-type: none">• Training to use microtome.• Prepared the Specimen of NASA-T1000 for ESEM Test (fiber-slicing method).
January 14	<ul style="list-style-type: none">• Prepared presentation for conference.• Completed ESEM test (fiber-slicing method) for NASA-T1000
January 21	<ul style="list-style-type: none">• Prepared presentation for conference.• Analyzed the NASA-T1000 strain and CTE data.
January 28	<ul style="list-style-type: none">• Electron diffraction pattern for NASA-T1000-HT-01, NASA-T1000-HT-02-1 and Amoco P55J2K.

2. Focus Areas

Electron diffraction pattern of NASA-T1000-HT-01, NASA-HT-02-1 and Amoco P55J2K.

3. Results of TEM for Electron diffraction pattern for

Electron diffraction pattern images were produced for NASA-T1000-HT-01, NASA-HT-02-1 and Amoco P55J2K fibers. NASA-T1000-HT-01 is heat-treated (the temperature unknown) and received from NASA in August 2001. NASA-HT-02-1 is also heat-treated (the temperature was quoted as 4800F) and was received from NASA in January 2002. The TEM results are presented in Figure 1-4.

We can observe that both the core area and the skin area of T1000 fibers are crystalline from Fig. 1-2. However the skin region displays more crystallinity than the core. In Fig 3, the EDP of the core sections for regular and heat-treated T1000 fibers are presented for comparison. Note that the core area of NASA-T1000 (as-received) is amorphous one, but both of heat-treated NASA-T1000 fibers are crystalline at the core area.

Fig 4.(Amoco-P55J2K that is pitch-based fiber) shows that both of core area and skin area have crystalline structure and the this crystalline structure is different from those observed in T1000 fibers..

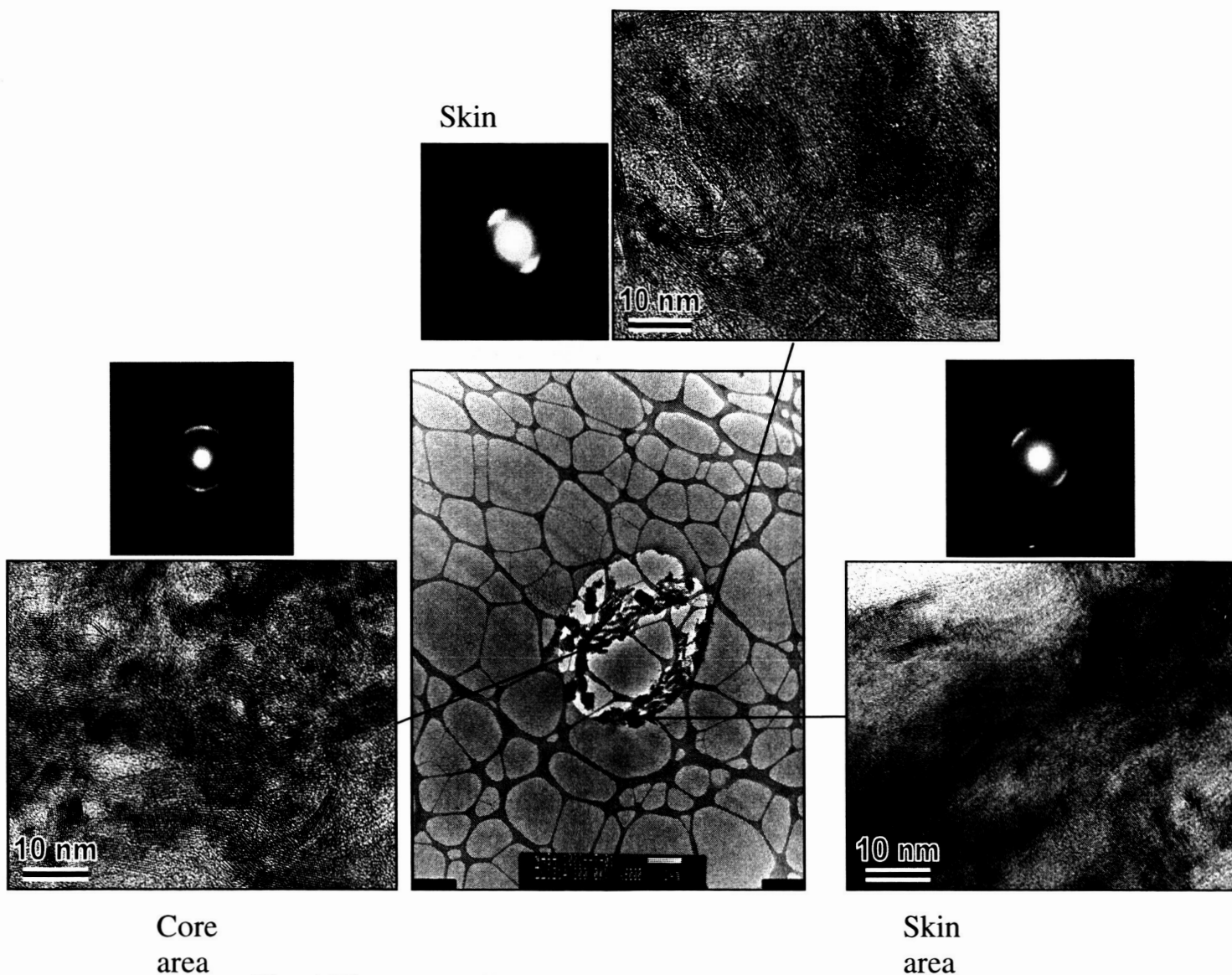


Fig. 1 Electron Diffraction Pattern for NASA 1000-HT-01 fibers

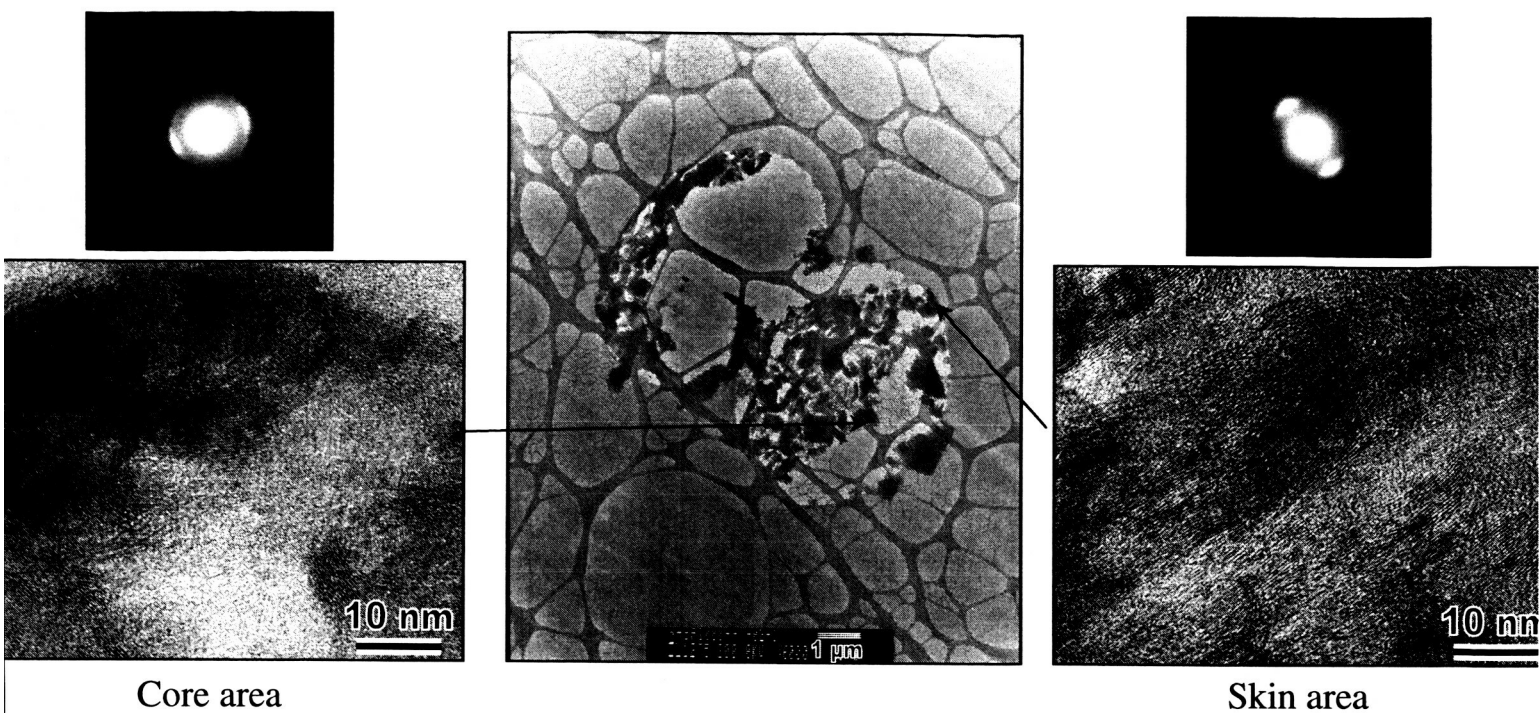
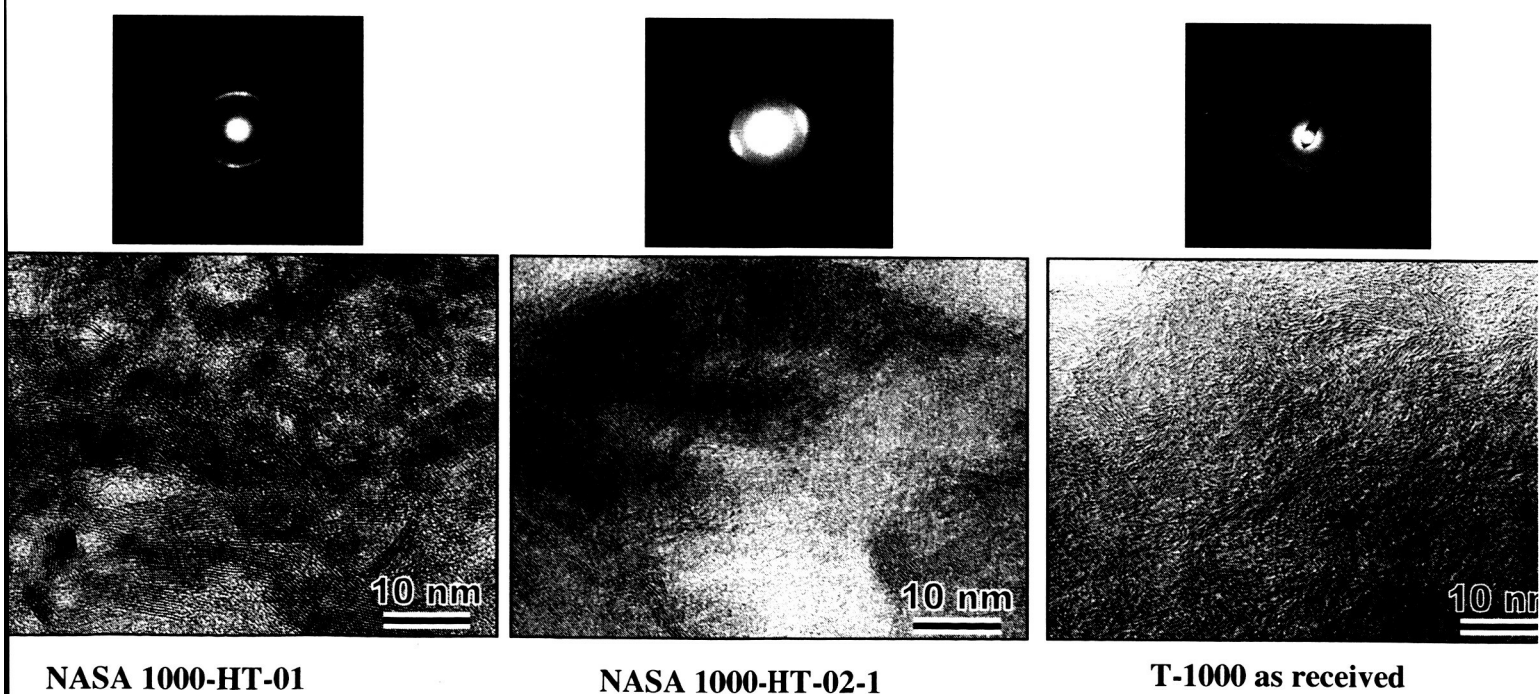


Fig. 2 Electron Diffraction Pattern for NASA 1000-HT-02-1 fibers

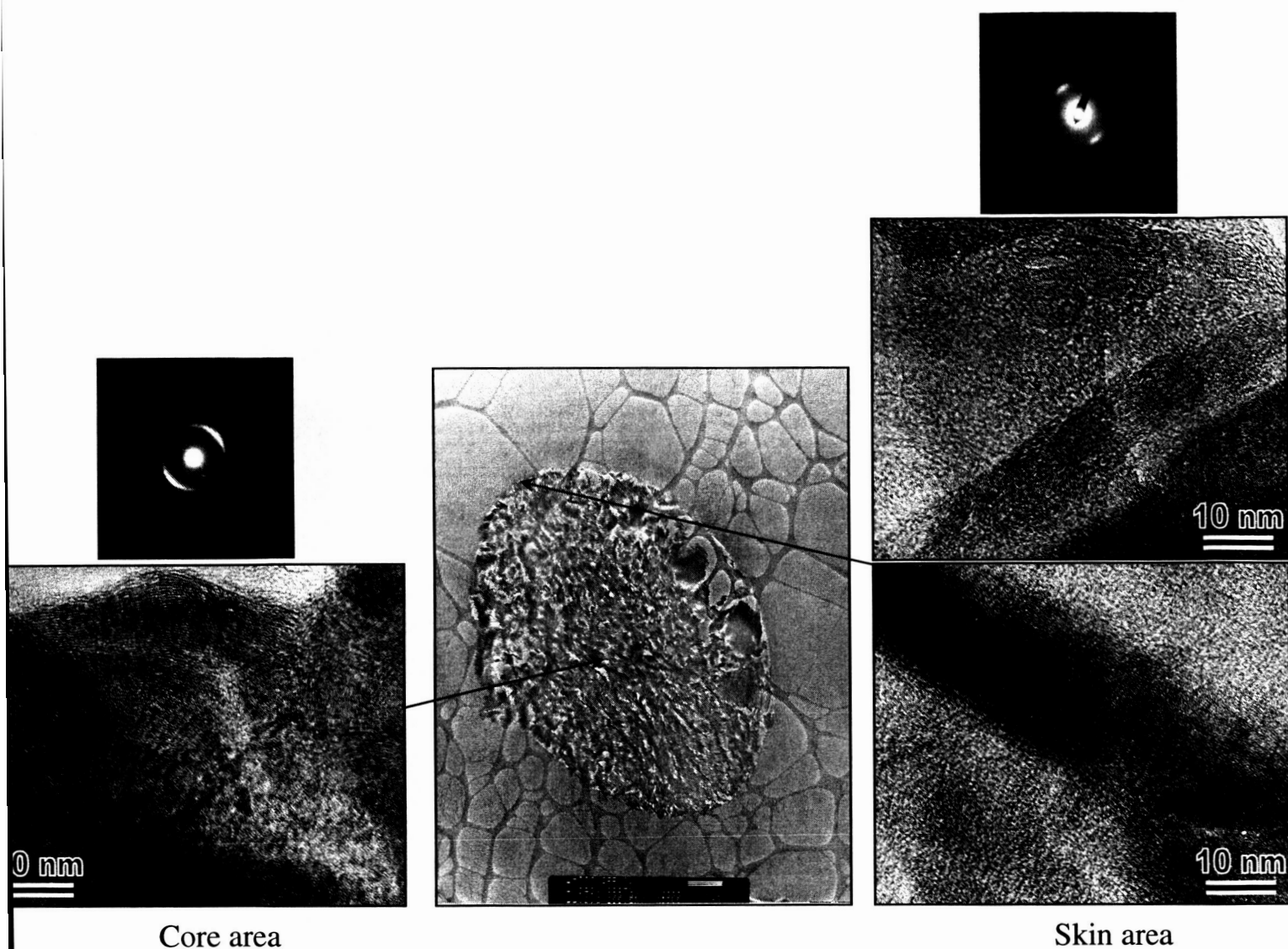


NASA 1000-HT-01

NASA 1000-HT-02-1

T-1000 as received

Fig. 3 Comparison of Electron Diffraction Patterns for core areas



Core area

Skin area

Fig. 4 Electron Diffraction Pattern for Amoco P55J2K fibers

4. Future work

- TEM test to get Electron diffraction pattern for NASA-T1000 (ESEM-Tested), and compare the results for NASA-T1000 fibers.
- Test the fixture for axial CTE measurement in ESEM.
- ESEM Test for NASA-T1000 using new method (fiber slicing method).

THERMOPHYSICAL ESEM & TEM CHARACTERIZATION of CARBON FIBERS

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Monthly Progress Report

February 2002

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Accomplishments

1. Monthly Summary

Week of	
February 3	<ul style="list-style-type: none">• Prepared the Specimens of NASA-T1000 fiber for ESEM Test (fiber-slicing method).• Completed ESEM test (fiber-slicing method) for NASA-T1000
February 10	<ul style="list-style-type: none">• Made FEM 3D model to calculate fiber deflection due to thermal expansion.• Completed ESEM test (fiber-slicing method) for NASA-T1000
February 17	<ul style="list-style-type: none">• FEM 3D model to estimate transverse CTE using optimization method.• Analyzed the NASA-T1000 strain and CTE data.
February 24	<ul style="list-style-type: none">• Preparations to incorporate fixture for axial CTE measurement in ESEM.

2. Focus Areas

- a) Fixture design for axial CTE measurement in ESEM.
- b) ESEM Test for NASA-T1000 using new method (fiber slicing method).
- c) FEM 3D model to estimate transverse CTE using optimization method.

3. Results

3.1 ESEM Test.

We are addressing few problems in our latest ESEM method (fiber slicing method). When we coat the specimen with gold and heat it to eliminate the epoxy used to hold it, we lose most of the fibers. Now, we are preparing many more fiber segments so that there will still be some left. In some of cases, we can not find a suitable fiber segment (ie with a pristine cross section without ovalization and cracking) to take a picture in the ESEM. During February, we tried four ESEM Tests with NASA-T1000 fibers. We have two successful tests where there were fibers intact for imaging. However, the images from one of these tests were unclear.

The strain, and CTE results for NASA-T1000 fiber tested in February are presented in Figure 3. 1 where two ramp-up data sets are included. Fiber specimens were coated with gold and they were 1 micro-meter long.

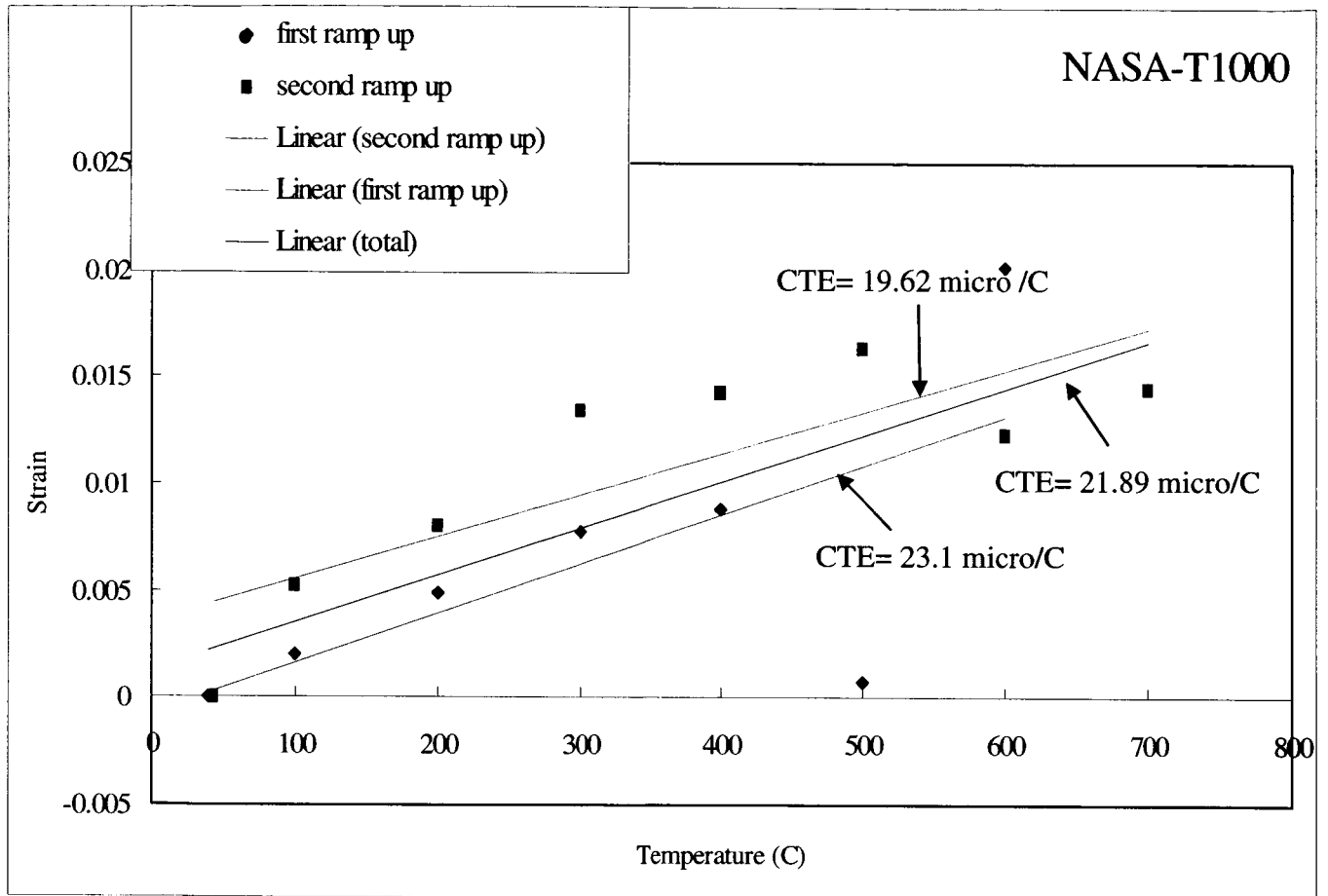


Fig. 3.1 ESEM Test result for NASA-T1000 (02/ 25/ 02 test)

Table 3.1 shows CTE values for each data set. CTE value of first ramp up data set from Jan. test shows much lower one.

CTE (*10 ⁻⁶)	01/22/02 test			02/25/02 test			Total
	First ramp up	Second ramp up	Total	First ramp up	Second ramp up	Total	
	2.7	28.7	15.7	23.1	19.6	21.9	

Table 3. 1 CTE values for NASA-T1000 fibers

Fig. 3. 2 shows CTE results that were calculated with three point strain data (ie;the average of strain at previous temperature, strain at temperature of interest and strain at the proceeding temperature) as a function of temperature. Before 400 C is reached, both data sets are very close each other. However, after 400 C, the CTE values are scattered. In this picture, average CTE values are the average between first ramp up and second ramp up for each temperature.

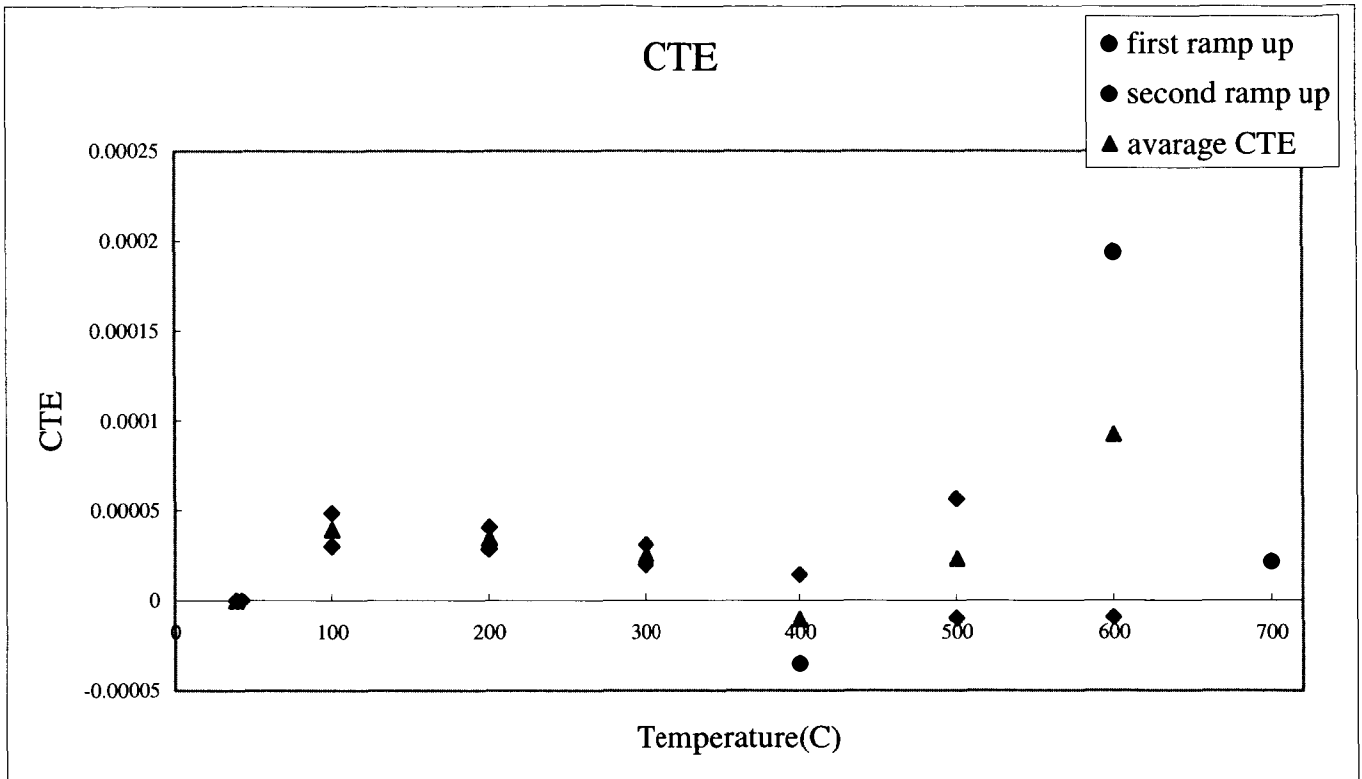


Fig. 3. 2 CTE using three-point-strain data (02/25/02 test).

3. 2 FEA Model for Optimization Method

A FEA model for the fibers was developed to calculate CTE through optimization method (first order method).

- Assumption-
 - Transversely isotropic material. (Longitudinal and transverse direction)
 - Cross-section is perfect circle. Therefore we can use diameter instead of perimeter
- Optimization variable
 - Objective Function = | Diameter from experiment – Diameter from FEA Model |

- Design Variable = CTE (transverse)

- Material properties (x-y plane is cross-section of fiber) --T300 fiber

(From G. Wagoner, R. Bacon. "Elastic constants and Thermal Expansion Coefficients of Various Carbon Fibers." In extended abstracts of 19th Biennial carbon conference, Penn State University PP. 296-297, 1987)

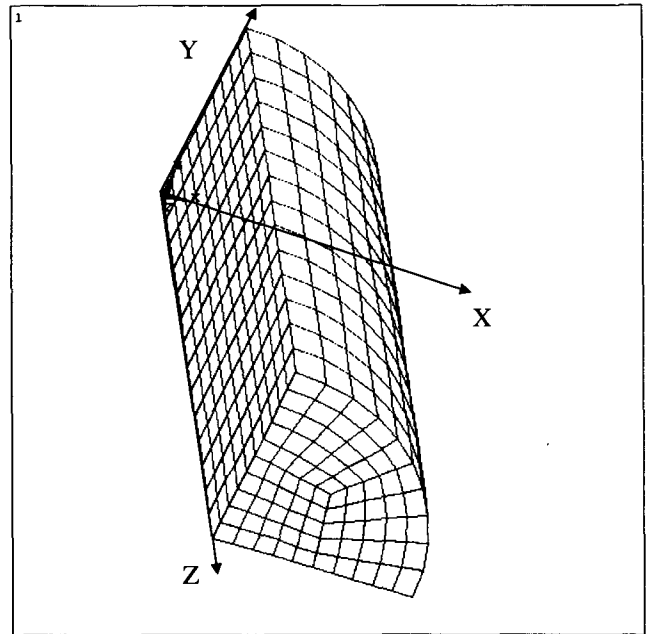
$E_x=E_y=10.3$ Gpa, $E_z=341$ Gpa,

$G_{xz}=G_{yz}=19.0$ Gpa, $G_{xy}=3.5$ Gpa,

$\nu_{zx}=0.13$,

CTE-z(longitudinal CTE)=-1.2 micro/C.

Fig. 3.3 FEA Coordinate system



In these three dimensional models, solid 73 element type of ANSYS (8 nodes with 6 degrees of freedom (U_x , U_y , U_z , Rot_x , Rot_y , Rot_z)) is used. Temperature differential is applied as a body force (from 20 C to 800 C). There are total of 1125 elements in this symmetric model.

- Procedure-

- Prepare FEA model to analyze fiber thermal behavior.
- Activate the optimization method (first order method, 20 iterations) and set tolerance
- Input experimental diameter at 800 C which is 0.256975E-05 (in this example, this is an arbitrary value that corresponds to an assumed transverse CTE of 7.5 E-6) and initial trial value (6.0 E-6) of design variable.
- Find design variable (transverse CTE) for the minimum value of objective function.

In this example, the CTE value from optimization method is 7.65E-6 within 1.7% of value of the 7.5E-6.

3.3 The Fixture for Axial CTE Measurement in ESEM.

Fig. 3.4 shows the schematic of the apparatus to measure axial CTE in ESEM. The fixture will use 2000lb capacity load cell to measure the load applied to the fiber bundle via a bolt tensioner. The fibers are wrapped around pins then held with a pinch plate to minimize end effects.

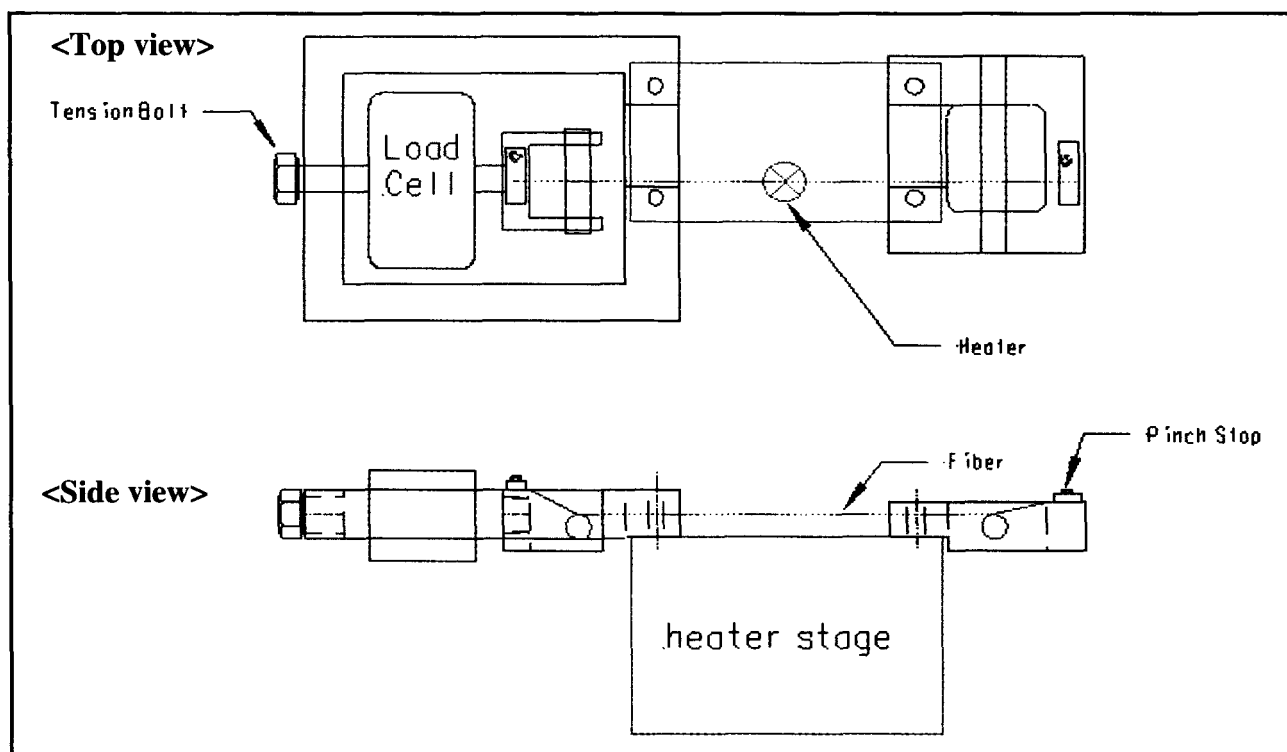


Fig. 3. 4 The schematic of the apparatus to measure axial CTE in ESEM

4. Future Work

- Continue TEM test to get Electron diffraction pattern for NASA-T1000 (ESEM-Tested), and compare the results
- Test the fixture for axial CTE measurement in ESEM.
- ESEM Test for NASA-T1000 with a shield to reduce electron disturbance to improve image clarity (also try without gold coating).

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Accomplishments

1. Monthly Summary

Week of	
March 3	<ul style="list-style-type: none">• Prepared the Specimens of NASA-T1000 fiber for ESEM Test (fiber-slicing method).
March 10	<ul style="list-style-type: none">• Completed ESEM test (fiber-slicing method) for NASA-T1000• Prepared the Specimens of NASA-T1000 fiber for ESEM Test (fiber-slicing method).• Made FEM 3D model to analyze stress field.
March 17	<ul style="list-style-type: none">• Prepared the Specimens of NASA-T1000 fiber for ESEM Test (fiber-slicing method).• FEM 3D model to analyze stress field.• Analyzed the NASA-T1000 strain and CTE data.• Incorporate fixture for axial CTE measurement in ESEM.
March 24	<ul style="list-style-type: none">• Completed ESEM test (fiber-slicing method) for NASA-T1000• Revising fixture for axial CTE measurement in ESEM.• Analyzed the NASA-T1000 strain and CTE data.

2. Focus Areas

- a) Incorporate and test the fixture for axial CTE measurement in ESEM.
- b) ESEM Test for NASA-T1000 using new method (fiber slicing method).
- c) FEM 3D model to analyze stress field for mini-specimen of fiber and epoxy.

3. Results

3.1 ESEM Test.

We found that the major reason for losing fiber segments is a strong wind that occurred in the chamber during vacuum of ESEM. In the previous tests (until 3/14/2002 test), we placed the fiber embedded in epoxy segments into the furnace and then, burned the epoxy off. Afterward, we transferred the remaining fibers into the ESEM. During the vacuum process, most of the fibers were lost. This month, we changed this process. Now, we put into ESEM fibers embedded in epoxy. We use the ESEM heating capability to slowly remove the epoxy before actually taking pictures. Subsequent to the removal of epoxy, then we start our regular procedure of "Ramp up" conditions. In March, we experimented taking pictures

without Gold coating and we were able to get very good images. Since applying to coating is another step where we are losing fibers, this month, we prepared samples without gold coating. We have two successful tests (3/14/2002 test and 3/26/2002 test) in where there were fibers intact for imaging. The strain, CTE results for NASA-T1000 fiber tested in March 14 are presented in Figure 3. 1 where three ramp-up data sets for one fiber are included. Fiber specimens are about 1 micro-meter long without gold coating.

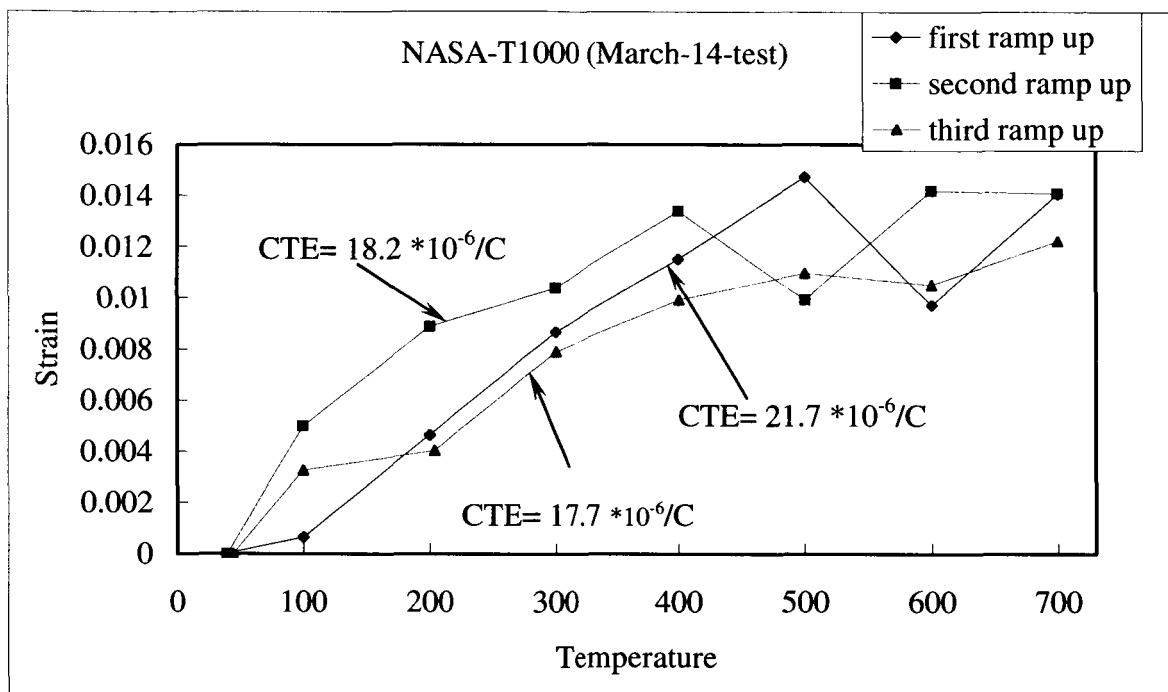


Fig. 3.1 ESEM Test result for NASA-T1000 (03/ 14/ 02 test)

The strain, CTE results for NASA-T1000 fiber tested in March 26 are presented in Figure 3. 2 where four ramp-up data sets for two fibers are included. Fiber specimens are about 1 micro-meter long without gold coating. At the March 26 test, we used ESEM heater to eliminate epoxy. Therefore, we were able to find much more intact fiber segments. We got images for three fibers at each temperature simultaneously. Since the images for third fiber were very ambiguous, we are reporting data from only two fibers.

CTE (*10-6)	1/22/02 test		2/25/02 test		3/14/2002 test			3/26/2002 test			
	1st ramp up	2nd ramp up	1st ramp up	2nd ramp up	1st ramp up	2nd ramp up	3rd ramp up	Fiber 1		Fiber 2	
	1st ramp up	2nd ramp up	1st ramp up	2nd ramp up	1st ramp up	2nd ramp up	3rd ramp up	1st ramp up	2nd ramp up	1st ramp up	2nd ramp up
	2.7	28.7	23.1	19.6	21.7	18.2	17.7	15.3	13.9	18.1	16.6

Table 3. 1 CTE values for NASA-T1000 fibers

Table 3.1 shows CTE values for each data set. These CTE values are the slope of linear fitting line for each strain-temperature data set. However, In Fig. 3.1 and Fig. 3. 2, note that

the shape for all data sets is nonlinear. Therefore, we chose to utilize various fitting options as presented in Fig. 3.3 and Fig 3. 5.

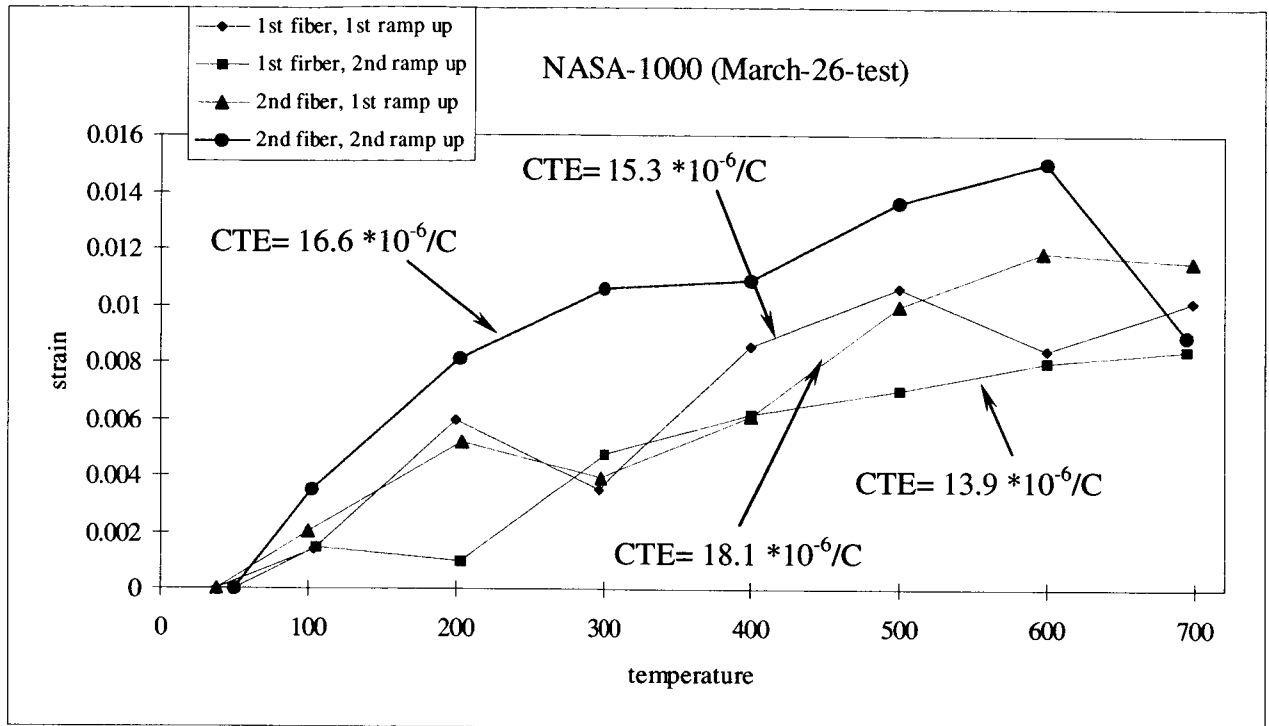


Fig. 3.2 ESEM Test result for NASA-T1000 (03/ 26/ 02 test)

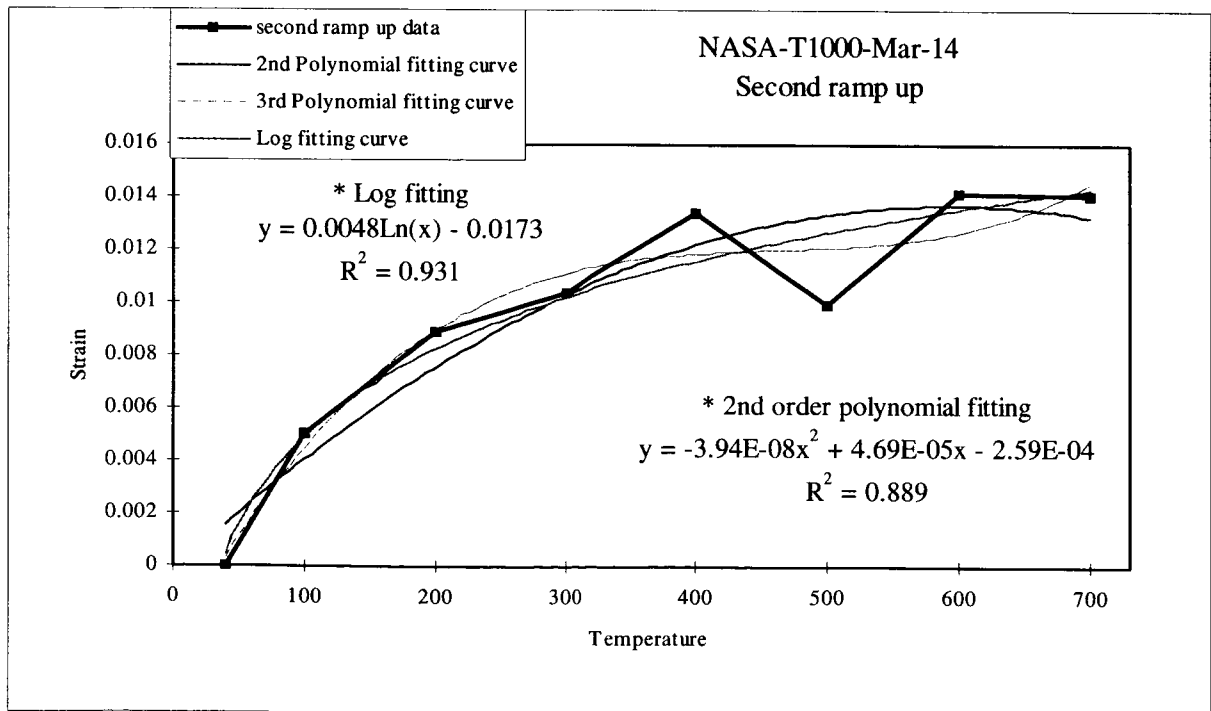


Fig. 3.3 various fitting for second ramp up data set (3/14/2002 test)

We calculated CTE values that were deduced by the derivative of the fitting curve [1] and are shown in Fig. 3. 4, Fig 3. 6 and Table 3. 2 where 3 point data points means the slope of linear fitting line using consecutive 3 strain data and temperature corresponding each temperature data.

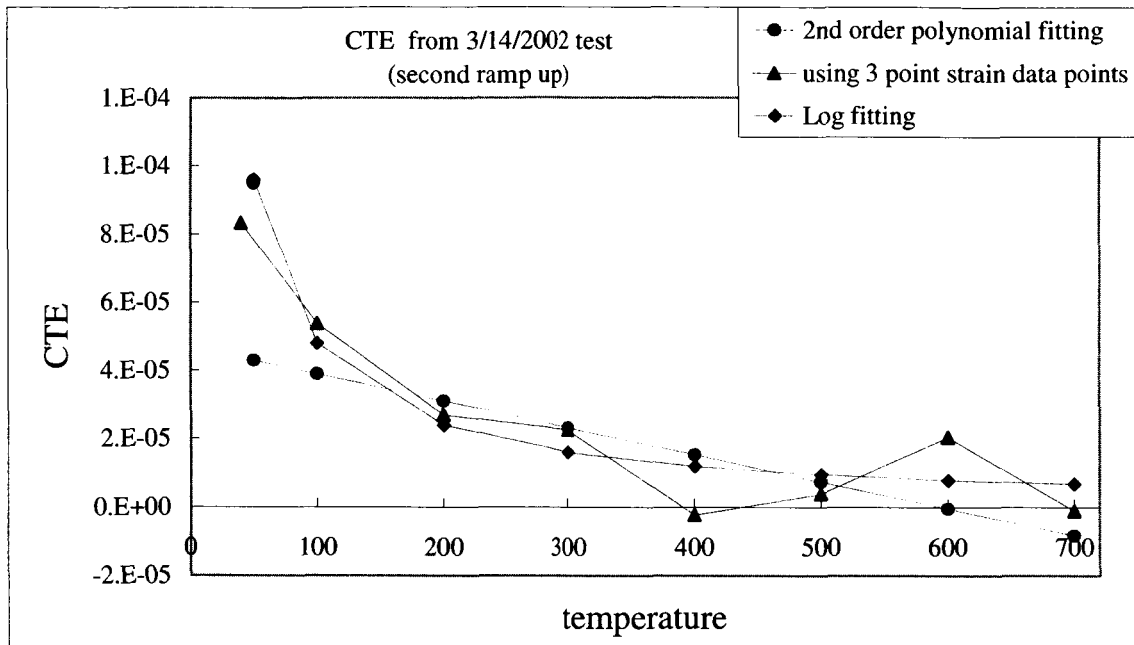


Fig. 3.4 comparison of CTE values for various fitting method for second ramp up data set (3/14/2002 test)

temp	CTE	
	2 nd order poly	Log
50	4.30E-05	9.60E-05
100	3.90E-05	4.80E-05
200	3.11E-05	2.40E-05
300	2.32E-05	1.60E-05
400	1.54E-05	1.20E-05
500	7.47E-06	9.60E-06
600	-4.23E-07	8.00E-06
700	-8.31E-06	6.86E-06

Table. 3. 2 comparison of CTE values for various fitting method (3/14/2002 test, second ramp up)

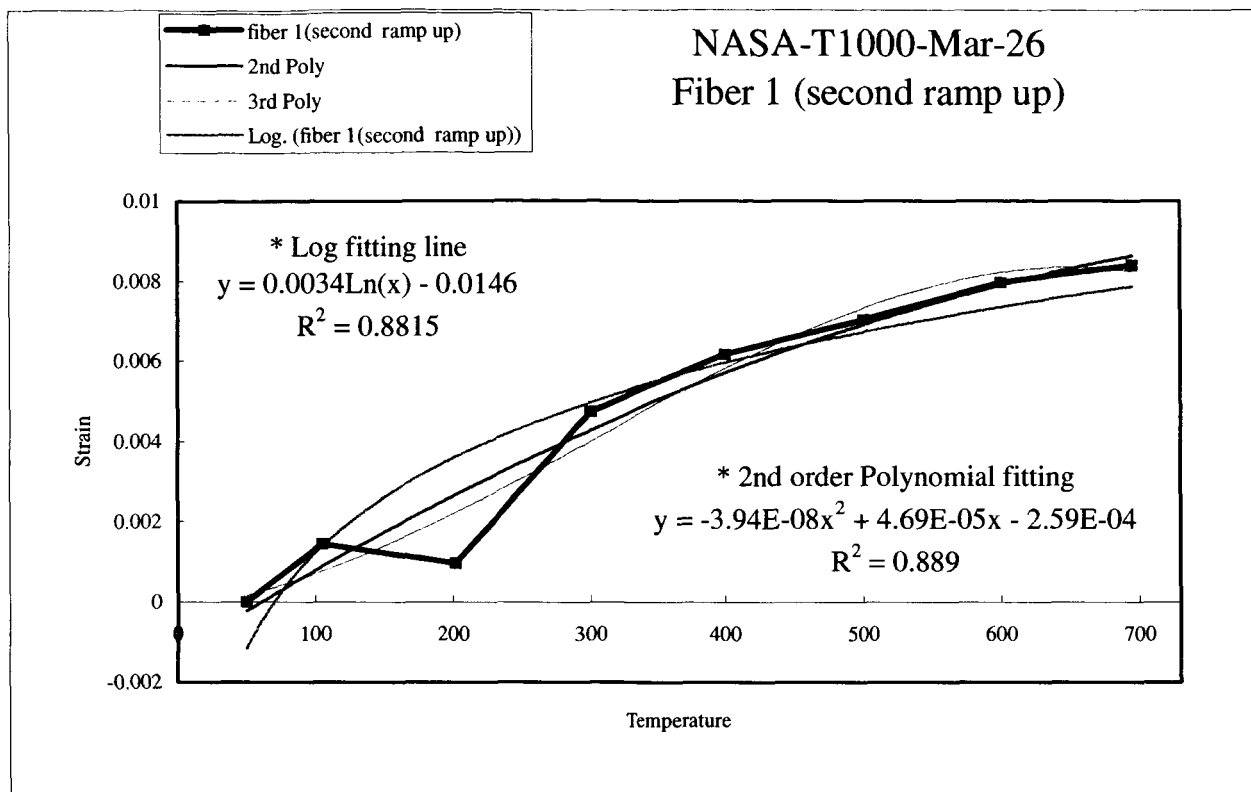


Fig. 3.5 various fitting for fiber 1, second ramp up data set (3/26/2002 test)

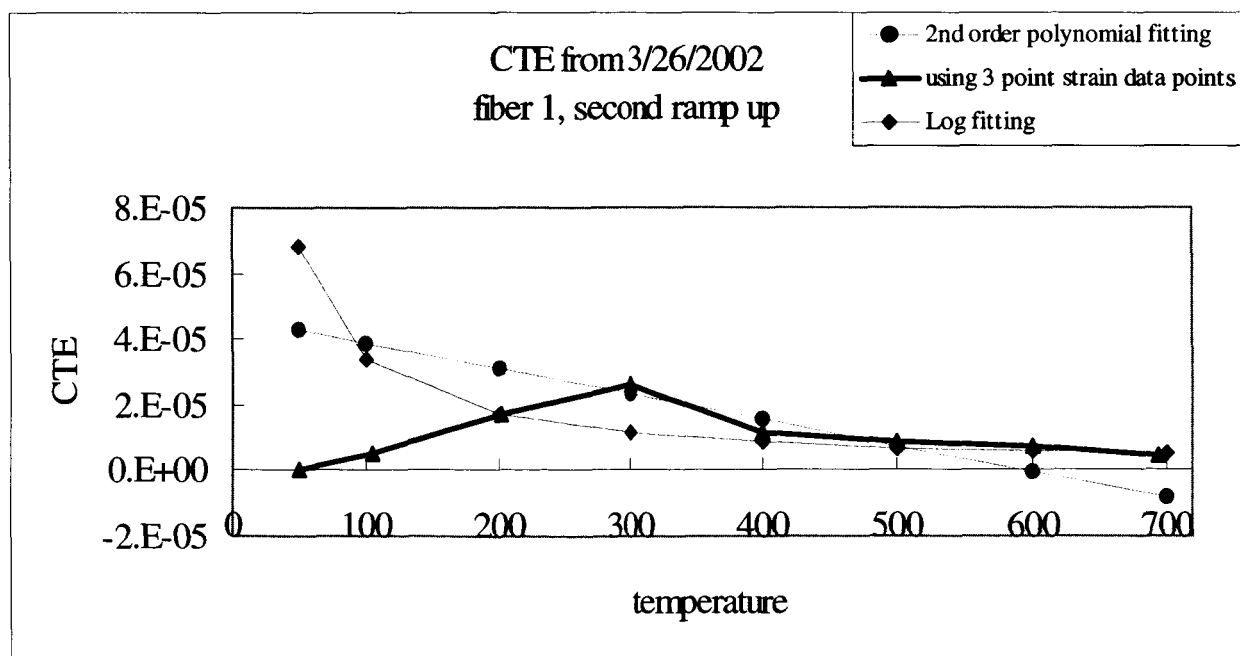


Fig. 3. 6 comparison of CTE values for various fitting method for fiber 1, second ramp up data set (3/26/2002 test)

3. 2 FEA Model for stress field due to thermal loading

A FEA model for the mini-specimen of fiber and matrix was developed to analyze stress field due to thermal loading.

- Assumption

- ◆ Transversely isotropic material for fiber. (Longitudinal and transverse direction).

- ◆ Isotropic material for matrix.

- Cross-section of fiber is a circle.

- Material property (x-y plane is cross-section of fiber) --T300 fiber

- Fiber: T300 fiber (From G. Wagoner, R. Bacon. "Elastic constants and Thermal Expansion Coefficients of Various Carbon Fibers." In extended abstracts of 19th Biennial carbon conference, Penn State University PP. 296-297, 1987)

: $E_x=E_y=10.3 \text{ GPa}$, $E_z=341 \text{ GPa}$, $G_{xz}=G_{yz}=19.0 \text{ GPa}$, $G_{xy}=3.5 \text{ GPa}$, $\nu_{zx}=0.13$, CTE

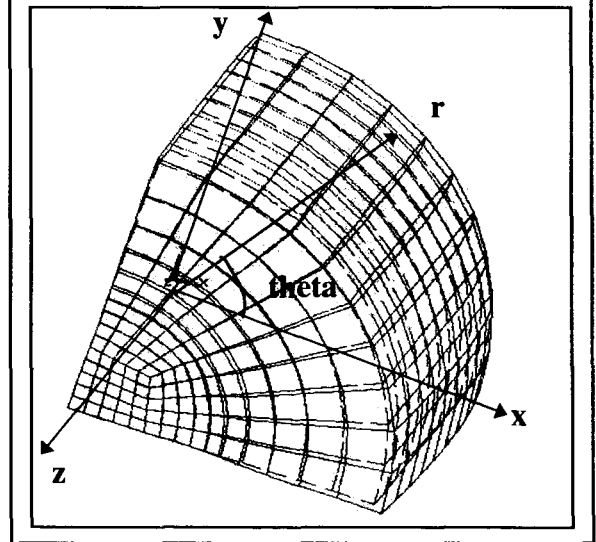
$z(\text{longitudinal CTE})=-1.2 \times 10^{-6}/^\circ\text{C}$. $\text{CTE}_x=\text{CTE}_y$ (Transverse CTE)= $7 \times 10^{-6}/^\circ\text{C}$

- ◆ Matrix: Silicon Carbide (From Dynamic -ceramic LTD): $E= 390 \text{ GPa}$, $\nu=0.24$,

$\text{CTE}= 3 \times 10^{-6}/^\circ\text{C}$

In this three dimensional models, solid 73 element type of ANSYS where 8 nodes with degrees of freedom (U_x , U_y , U_z , Rot_x , Rot_y , Rot_z) is used. Temperature is applied as a body force ($\Delta T = 280^\circ\text{C}$). There are total of 1250 elements with symmetry in the model. The original and deformed shape is presented in Fig. 3. 7. Fig. 3. 8 shows stress fields for stress_r. Maximum stress stress_r occurs at near the fiber interface as shown in Fig. 3.8. Fig. 3. 10 and Fig 3.11 shows stress fields for stress_{theta}.

Fig. 3. 7 Original and deformed shape



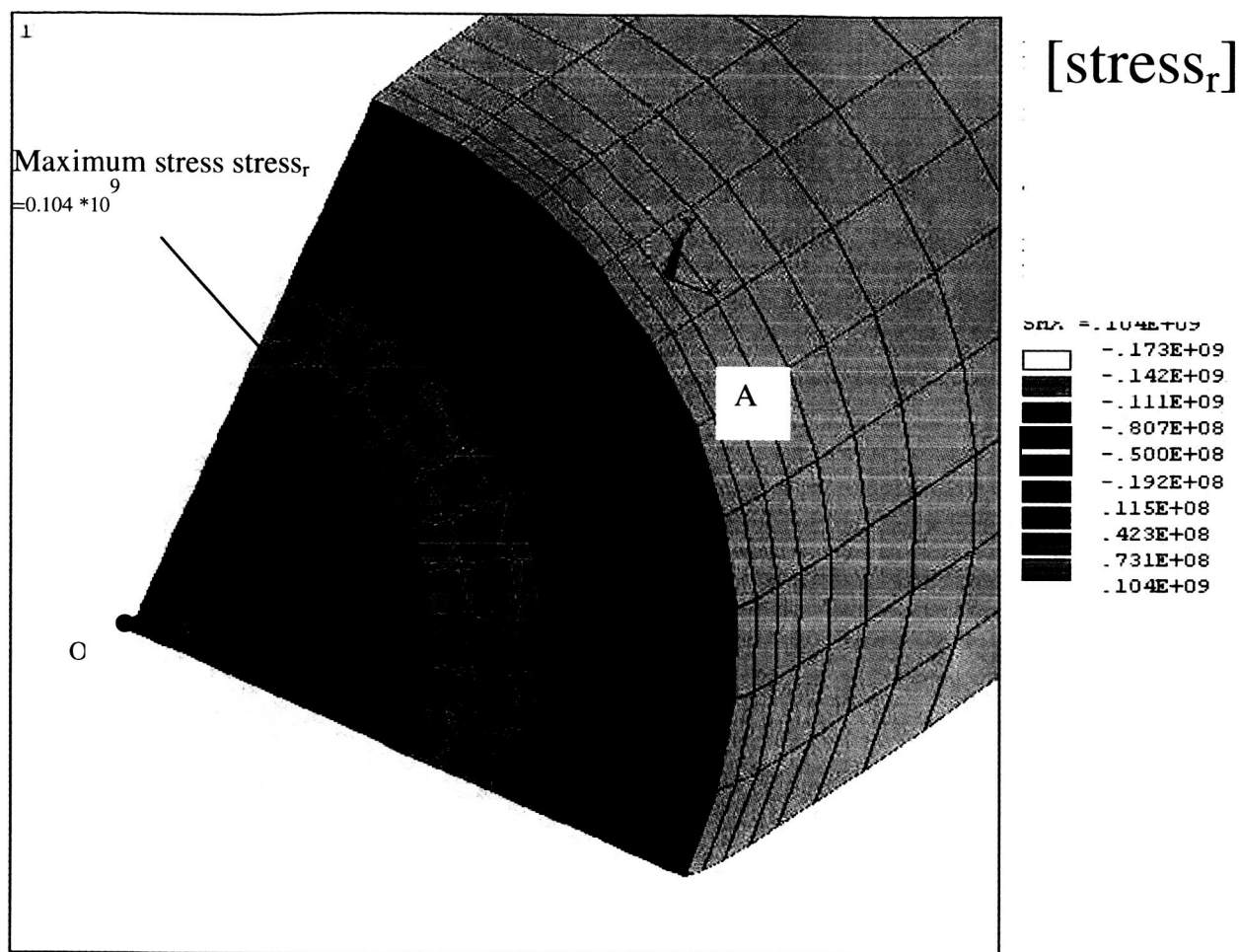


Fig. 3.8 stress field (stress_r)

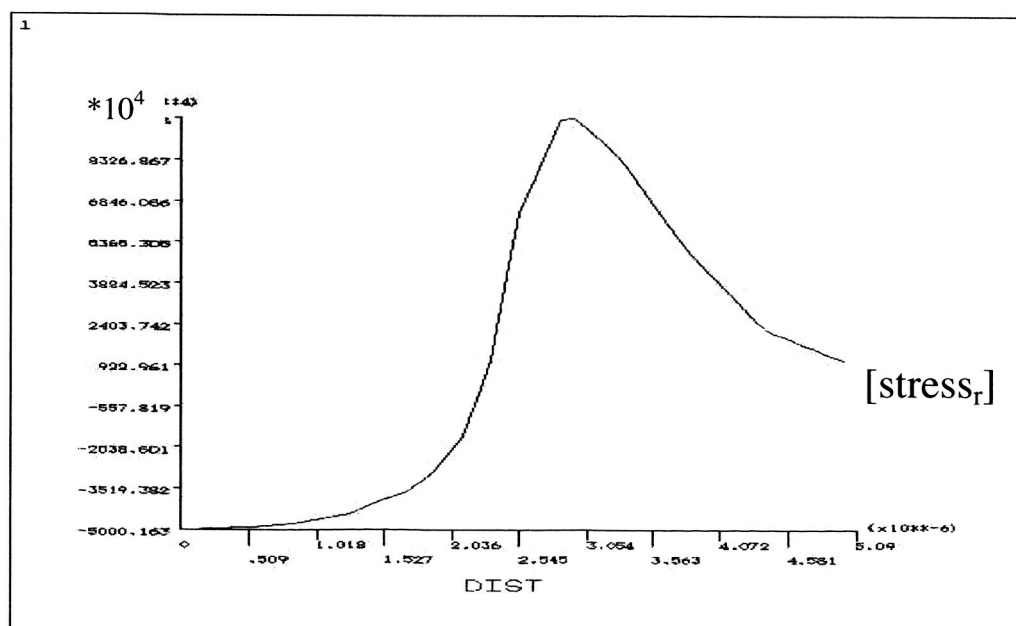


Fig 3.9
stress(STRESS_r)
graph
through line
OA

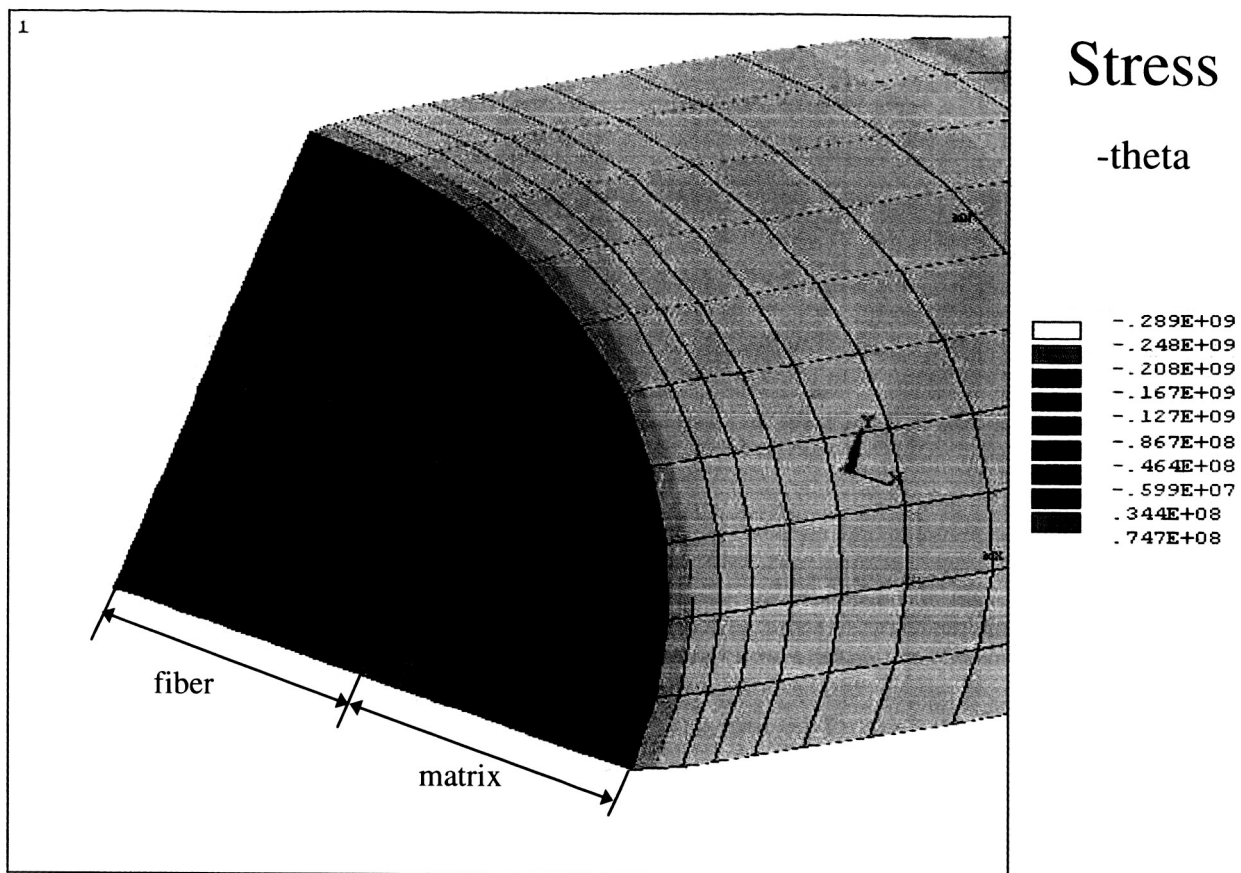


Fig. 3.10 stress field (stress_{θ})

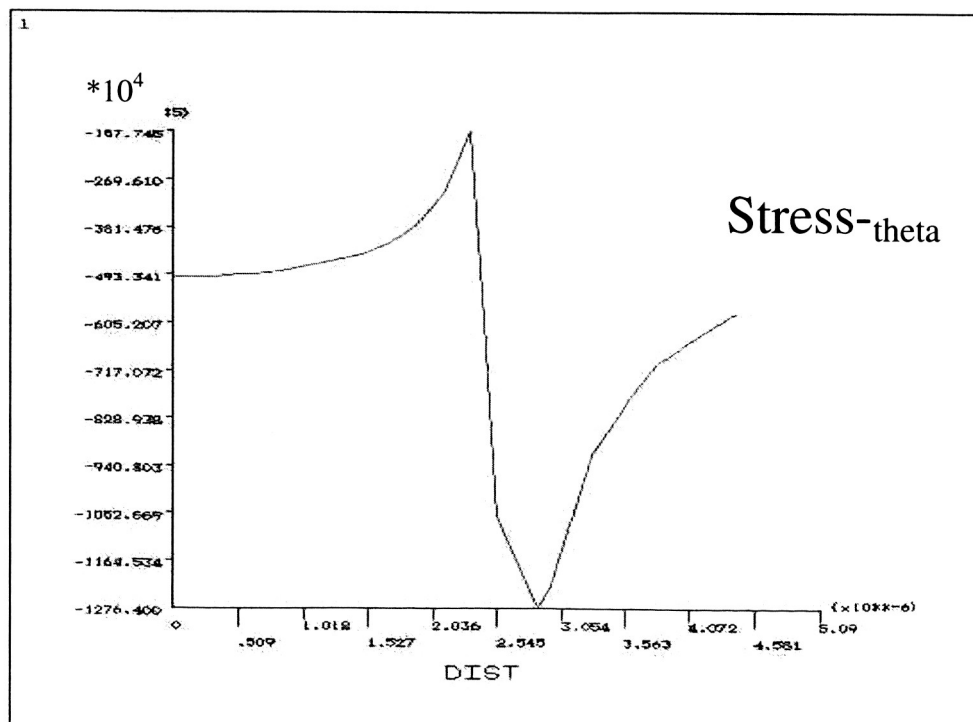
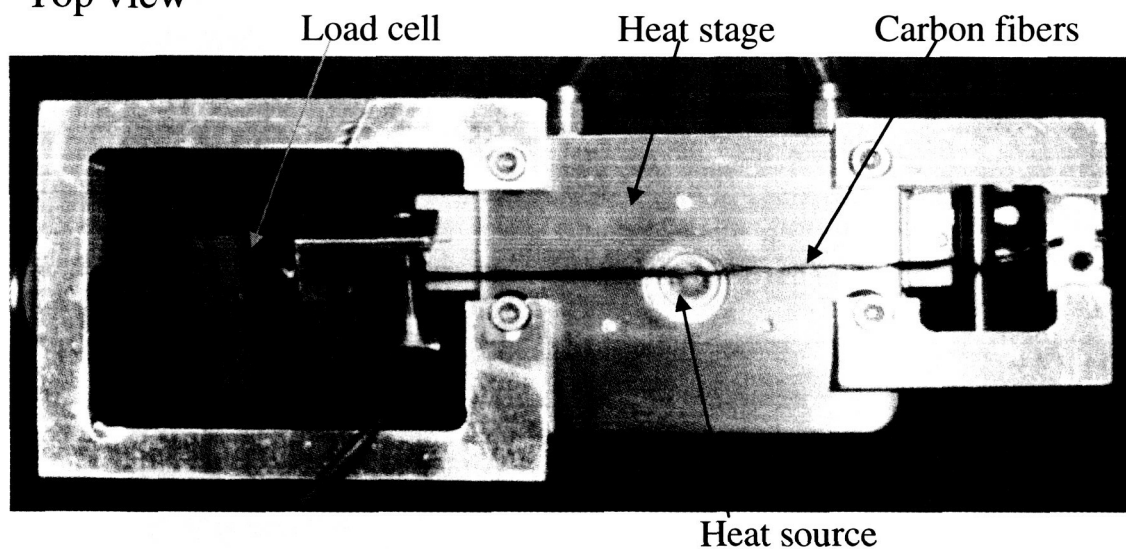


Fig 3.11 stress- θ graph through line OA

3.3 The Fixture for Axial CTE Measurement in ESEM.

Fig. 3.5 shows the schematic of the apparatus to measure axial CTE in ESEM. CTE fixture device uses 2000lb capacity load cell to measure the load applied to the fiber bundle via a bolt tensioner. The fibers are wrapped around pins then held with a pinch plate to minimize end effects. We incorporated this device in ESEM. Every thing was working well. But, The position of Load cell is too high and we were able not to get the suitable distance from electron Probe of ESEM to specimen to take a clear image.

Top view



Side view

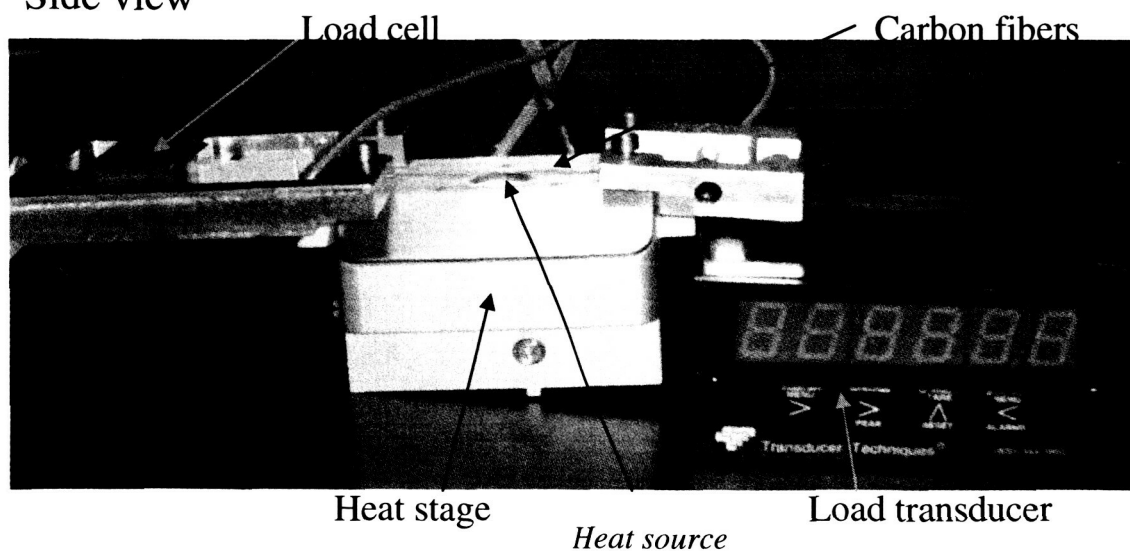


Fig. 3. 12 The apparatus to measure axial CTE in ESEM

4. Future Work

- a) TEM test to get Electron diffraction pattern for NASA-T1000 (ESEM-Tested), and compare the results for NASA-T1000 fibers.
- b) Test the fixture for axial CTE measurement in ESEM.
- c) ESEM Test for NASA-T1000 for 5 fibers and compare temperature-strain data each other.

Reference :

- [1] M. Trinqucoste, J. L. Carlier, A.Derre, P. Delhaes and P. Chadeyron," High Temperature Thermal and Mechanical Properties of High Tensile Carbon Single Filaments", Carbon Vol. 34, No. 7, pp. 923-929, 1996

THERMOPHYSICAL ESEM & TEM CHARACTERIZATION of CARBON FIBERS

**CTE, SPECTROSCOPY and ROUGHNESS STUDIES at
HIGH TEMPERATURES
(Grant NAG 8 –1802)**

Monthly Progress Report

April 2002

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Accomplishments

1. Monthly Summary

Week of	
April 1	<ul style="list-style-type: none">• Prepared the Specimens of NASA-T1000 fiber for ESEM Test (fiber-slicing method)• Conducted ESEM test using argon gas.
April 8	<ul style="list-style-type: none">• Literature review for oxidization of carbon fiber.• Testing fixture for axial CTE measurement in ESEM.• ESEM test (fiber-slicing method) for NASA-T1000
April 15	<ul style="list-style-type: none">• FEM 3D model to analyze stress field for carbon fiber/epoxy matrix.• Calibrating heat stage in ESEM using reference materials.
April 22	<ul style="list-style-type: none">• ESEM Test -carbon fibers embedded into epoxy(in place).• Revising fixture for axial CTE measurement in ESEM.

2. Focus Areas

- a) Literature search details on degradation of carbon fibers by oxidization.
- b) Calibrating heat stage in ESEM by reference materials
- c) Incorporate and test the fixture for axial CTE measurement in ESEM.

3. Results

3.1 Degrading of Carbon fibers.

In the previous tests (3/26/2002 test), we put into ESEM fibers embedded in epoxy. We use the ESEM heating capability under atmosphere to slowly remove the epoxy before actually taking pictures. Subsequent to the removal of epoxy, then we start our regular procedure of "Ramp up" conditions. However, this epoxy eliminating method exposes fibers to air under high temperature and may cause degradation [1],[2]. Fig. 3. 1 from [1] shows that weight loss starts at about 400°C and from 500 to 800°C, the weight loss is sharp for three fibers in atmosphere: I (Kureha isotropic pitch-based carbon fibers), IG (Kureha

isotropic pitch-based carbon fibers graphitized at 2700°C) and P-25 (Amoco mesophase pitch-based carbon fibers)) [1].

To prevent potential oxidation, we tried ESEM test in argon gas instead of occasional flushing with water vapor. However, the images from test using argon gas were very unclear. We can not adopt the test method by argon gas.

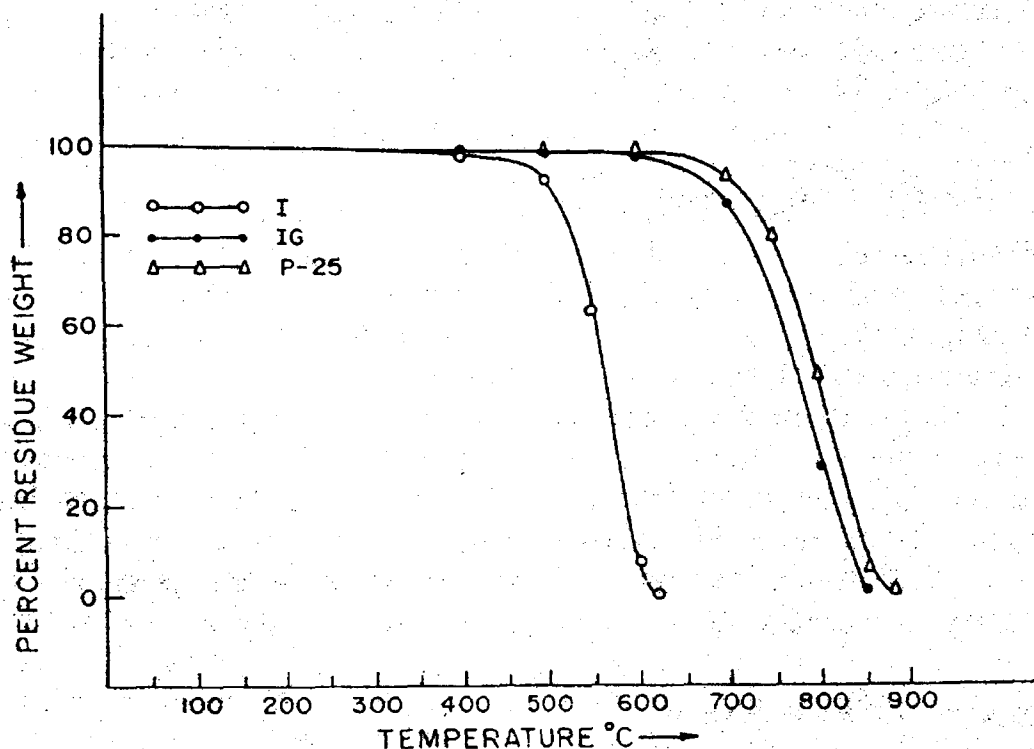
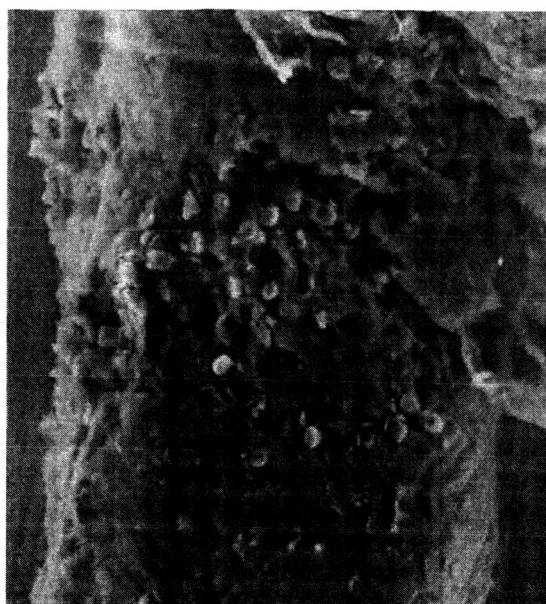


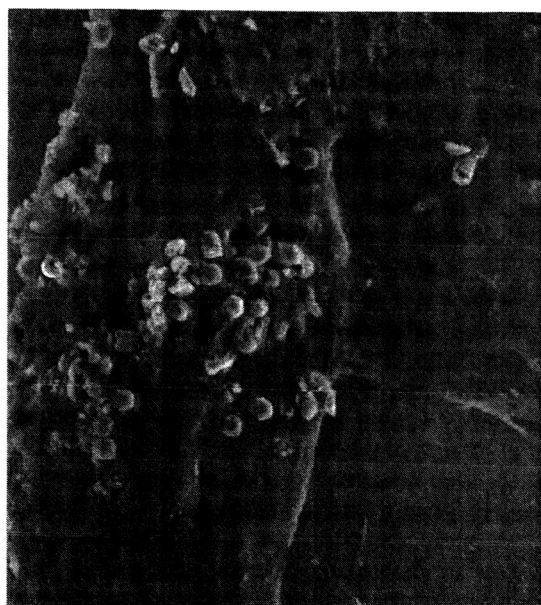
Fig. 3. 1 Weight loss by temperature rising from [1]

3. 2 Observing fiber and epoxy

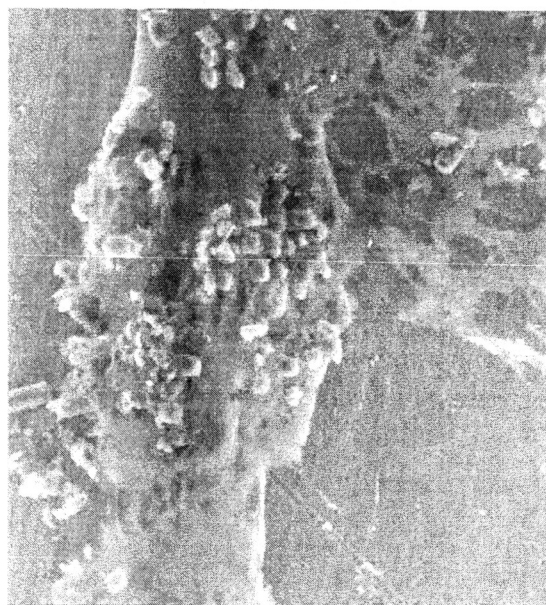
As an alternate, we studied the fiber-epoxy specimen without removing the epoxy in ESEM. Fig 3.2 shows the change of the specimen of carbon fiber (NASA-T1000) embedded into epoxy according to rising temperature. Fibers embedded into epoxy are shown in (a). Epoxy was starting melting at about 260C in (b). Until about 600C, epoxy kept melting. However, suddenly epoxy and fiber was moving fast at around 650C and at about 700C, most of fiber and epoxy were gone in (d). We did this test twice and observed the same phenomena. It is possible that electronic charging causes the disappearance of carbon fiber and epoxy because the thickness of specimen is relatively small.



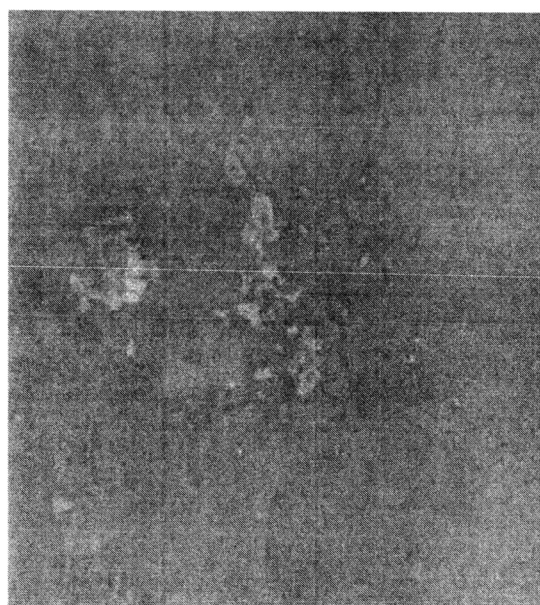
(a) 130 C



(b) 360 C



(c) 610 C



(d) 730 C

Fig 3. 2 ESEM images for Carbon fiber and epoxy matrix specimens for each temperature.

3. 3 Calibration of heating stage in ESEM

Table 3. 1 Calibration of ESEM Heat sensor

Material	Reference melting temperature (C)	Measured melting temperature (C)	Difference
Tin	232	195	-37
Pb	327	275	-52
Zinc	419	355	-64
AL	660	679	19

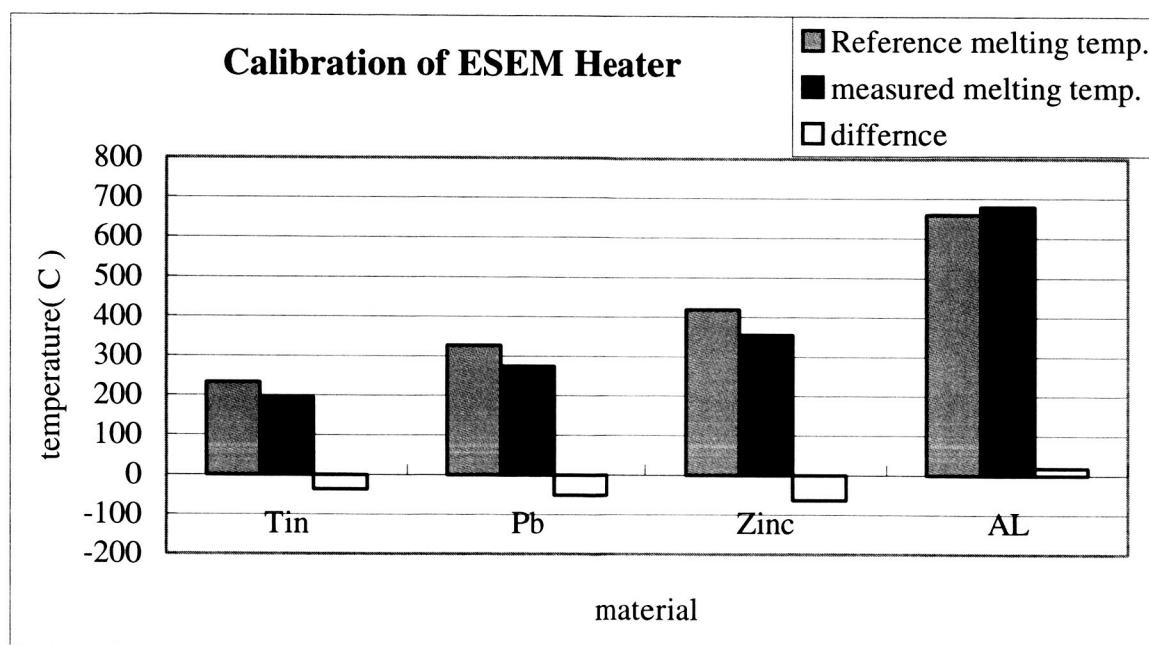


Fig. 3.3 Calibration of ESEM Heat sensor.

As a part of our continuous effort to assure the accuracy of heating environment, we looked at the melting temperatures of known metallics for calibration. Table 3.1 and Fig 3.3 show the result of calibration of ESEM heat sensor. We use reference materials to test that the temperature from heat stage of ESEM is right or not. We measured the melting temperature of reference material from heat stage of ESEM and compare this temperature with real melting temperature of reference material in Fig 3.3.

3. 4 Stress fields for carbon fiber/epoxy matrix by FEA Model

A FEA model for the mini-specimen of fiber and matrix was developed to analyze stress field due to thermal loading.

- Assumption

- ◆ Transversely isotropic material for fiber. (Longitudinal and transverse direction).
- ◆ Isotropic material for matrix.
- Cross-section of fiber is a circle.

- Material property (x-y plane is cross-section of fiber) --T300 fiber

- Fiber: T300 fiber (From G. Wagoner, R. Bacon. "Elastic constants and Thermal Expansion Coefficients of Various Carbon Fibers." In extended abstracts of 19th Biennial carbon conference, Penn State University PP. 296-297, 1987)

: $E_x=E_y=10.3 \text{ Gpa}$, $E_z=341 \text{ Gpa}$, $G_{xz}=G_{yz}=19.0\text{Gpa}$, $G_{xy}=3.5\text{Gpa}$, $V_{zx}=0.13$, CTE.

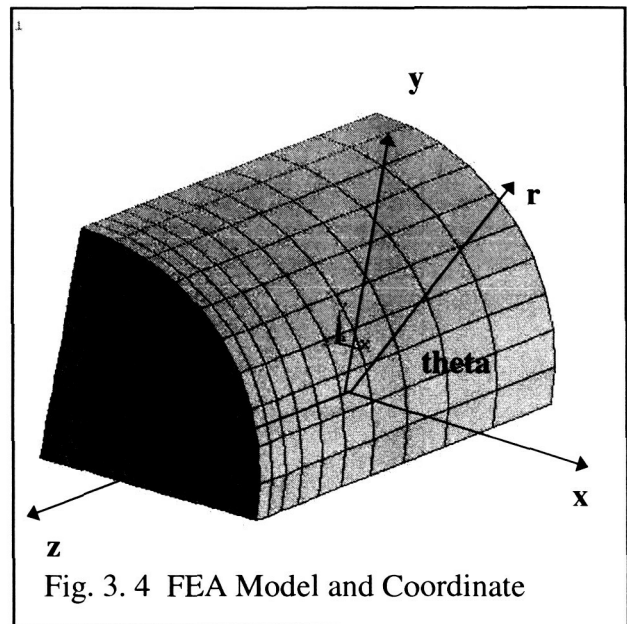
$z(\text{longitudinal CTE})=-1.2 \cdot 10^{-6}/\text{C}$. $\text{CTE}_x=\text{CTE}_y$ (Transverse CTE)= $7 \cdot 10^{-6}/\text{C}$

- ◆ Matrix: Epoxy Resin (From Stress Analysis of fiber-reinforced composite material):

$E= 2.4 \text{ Gpa}$, $G_{xy}=1.7 \text{ Gpa}$, $\text{CTE}= 60 \cdot 10^{-6}/\text{C}$

In this three dimensional models, solid 73 element type of ANSYS where 8 nodes with degrees of freedom (U_x , U_y , U_z , Rot_x , Rot_y , Rot_z) is used.

Temperature is applied as a body force ($\Delta T = 300^\circ\text{C}$). There are total of 1250 elements with symmetry in the model. The original and model is presented in Fig. 3. 4. Fig. 3. 5 shows stress fields for stress_r . Maximum stress stress_r occurs at near the fiber interface as shown in Fig. 3.5. Fig. 3. 6 shows stress field along to line a for stress_r .



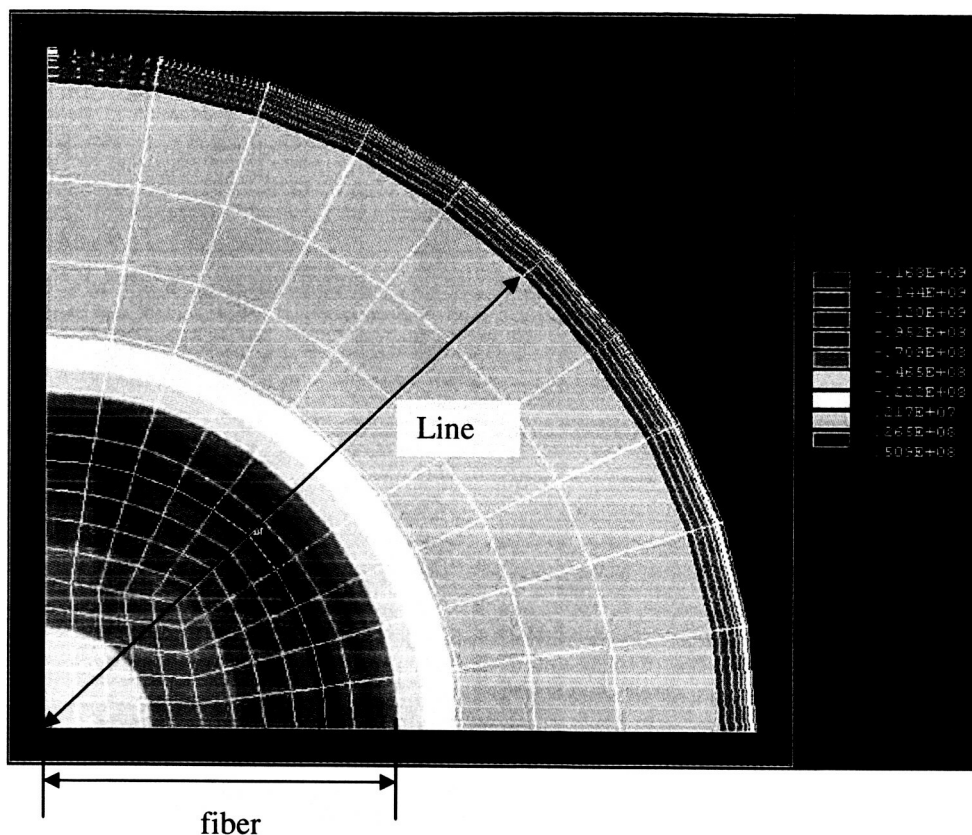
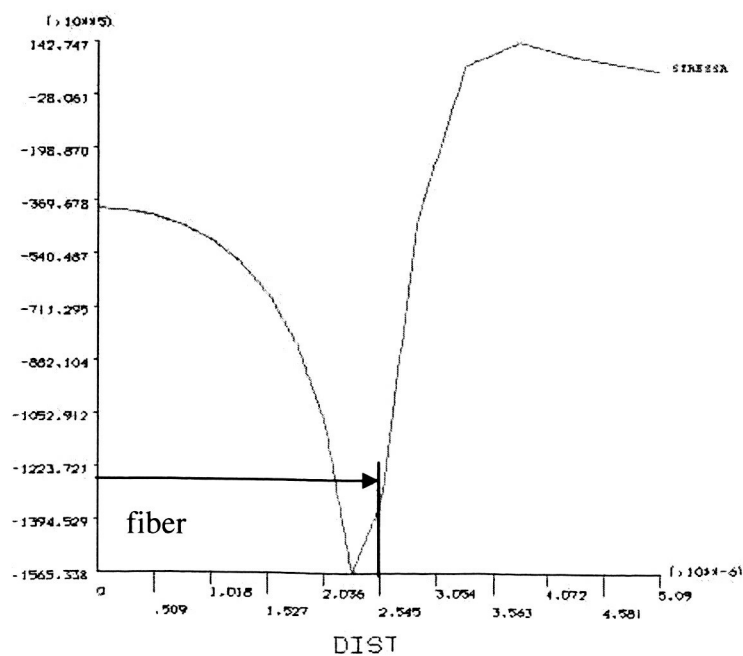


Fig 3.5 stress field for stress-radial

1

**Fig 3.6 Stress field
along to line**

A



3.5 The Fixture for Axial CTE Measurement in ESEM.

We changed design a little and incorporated fixture device in ESEM. Every thing was working well. However, the position of Load Cell is still too close to the heat sensor. Our machine shop is working with it now.

References

- [1] Deborah D. L. Chung. 1994 Carbon Fiber Composites. 75-77
- [2] Jean-Baptiste DONNET, Tong Kuan Wang, Serge REBOUILLAT and Jimmy C. M. PENG. 1998 Carbon Fibers. 3 ed. 163-170

THERMOPHYSICAL ESEM & TEM CHARACTERIZATION of CARBON FIBERS

**CTE, SPECTROSCOPY and ROUGHNESS STUDIES at
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Monthly Progress Report

December 2002

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Monthly Summary

December was dedicated to assuring our TEM work was repeatable in all aspects. At present we are preparing our presentation for Cape Canaveral CMC³ meeting and putting a draft for the journal article.

1A. Transverse CTE measurements in Environmental TEM

We continued our observations with TEM and looked at three additional T1000 fibers. Measurements along 15 different locations on each fiber were taken and the subsequent CTE values are provided in below. This indicates that potentially there is a variation from fiber to fiber.

T1000 CTE

1	5.63E-06	8.78E-06	1.21E-05
2	5.53E-06	8.31E-06	9.91E-06
3	5.32E-06	7.93E-06	1.09E-05
4	6.05E-06	7.74E-06	1.26E-05
5	7.16E-06	8.82E-06	1.26E-05
6	5.13E-06	7.29E-06	1.04E-05
7	5.50E-06	8.21E-06	1.19E-05
8	4.58E-06	7.90E-06	1.25E-05
9	5.90E-06	8.78E-06	1.26E-05
10	4.58E-06	7.42E-06	1.09E-05
11	4.76E-06	7.62E-06	1.26E-05
12	4.76E-06	7.95E-06	1.02E-05
13	4.22E-06	8.13E-06	1.04E-05
14	5.87E-06	7.71E-06	1.24E-05
15	4.17E-06	7.91E-06	1.22E-05
Average	5.28E-06	8.03E-06	1.16E-05

Similarly three P55 fibers were studied along 8 different locations within each negative taken in TEM. This time there is only a slight variation in CTE calculations from fiber to fiber. We attribute this narrow range to smooth surface of these fibers.

P55 Data

1	2.92E-06	3.25E-06	3.47E-06
2	3.56E-06	2.97E-06	3.47E-06
3	3.24E-06	3.52E-06	3.70E-06
4	3.23E-06	2.98E-06	3.40E-06
5	2.91E-06	3.25E-06	3.72E-06

6	3.56E-06	3.51E-06	3.96E-06
7	3.56E-06	3.79E-06	3.97E-06
8	3.24E-06	3.52E-06	3.47E-06
Average	3.28E-06	3.35E-06	3.65E-06

The results from four different IM7 fibers are also very close just like the P55 data. Note that IM7 also has a smooth surface. The measurements were taken at 15 and 8 different locations in respective fibers.

IM7 Data

1	4.05E-06	3.81E-06	5.81E-06	6.24E-06
2	3.92E-06	3.95E-06	5.22E-06	5.32E-06
3	3.92E-06	3.79E-06	4.26E-06	5.23E-06
4	4.01E-06	3.67E-06	5.71E-06	5.23E-06
5	4.01E-06	4.66E-06	5.52E-06	5.03E-06
6	4.18E-06	4.24E-06	3.86E-06	4.72E-06
7	4.32E-06	4.38E-06	4.45E-06	4.62E-06
8	4.32E-06	3.69E-06	4.06E-06	4.62E-06
9	4.32E-06	4.52E-06	--	--
10	4.45E-06	5.66E-06	--	--
11	4.45E-06	5.52E-06	--	--
12	4.20E-06	5.80E-06	--	--
13	4.02E-03	5.09E-06	--	--
14	4.03E-03	4.94E-06	--	--
15	4.05E-03	4.95E-06	--	--
Average	4.21E-06	4.58E-06	4.86E-06	5.13E-06

When we compare the data obtained via ESEM to that obtained in TEM, we note that the latter results are superior both in resolution during the actual measurements on the negatives as well as the repeatability in specimen preparation. Thus we would like to offer that the TEM measurements at present are closer to reality. As displayed below, ESEM evaluations are an order of magnitude higher.

